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# EXAMINATION OF COMMERCIAL HYPOPHOSPHITE PREPARATIONS.

By Frank X. Moerk, Ph.G.

Contribution from the Chemical Laboratory of the Philadelphia College of Pharmacy. No. 112.

In connection with other contributions on the *hypophosphites*, published in the American Journal of Pharmacy during the last few years, it was thought desirable to embrace an examination of the more soluble hypophosphite preparations as found in the Philadelphia market. The object was to ascertain the quantity of hypophosphites in preparations the formulas of which are kept secret; in others to make comparison of the quantity found and that claimed to be present, and lastly to allow a comparison to be made of these several preparations with those of the U. S. Pharmacopœia and the National Formulary.

The basic constituents were only qualitatively tested for; the metallic constituents generally being tested for in the residue after ignition. The alkaloids were extracted by diluting the preparations with water, rendering alkaline with sodium hydrate and agitating with chloroform; a little of the chloroform residue with sulphuric acid and a fragment of potassium bichromate revealed the presence or absence of *strychnine;* a larger portion of the residue dissolved in dilute sulphuric acid by its fluorescence, by the thalleioquin reaction, and by yielding a white precipitate with ammonia, readily soluble in excess, indicated *quinine.*

Of the several methods for the estimation of hypophosphites the one with mercuric chloride only was available since the other methods in presence of organic substances, like sugar, etc., give erroneous results. Having quite a number of these determinations to make it was found to be of advantage to ascertain the mercurous chloride

produced by a volumetric method instead of drying at 100° C. and weighing. All results are expressed in terms of *free hypophosphorous acid*, since the basic constituents varied in the several preparations. From 2–5 grams (in the first determination the smaller quantity was taken, and if the preparation was found to be *weak*, the larger quantity was taken in the duplicate) were diluted with 25 cc. water, 1 cc. concentrated hydrochloric acid and 25 cc. of a cold saturated solution of mercuric chloride added; after heating for one hour in a water-bath at 100° C., the separated mercurous chloride was filtered off, thoroughly washed with boiling water, allowed to drain, dropped (filter and precipitate) into a glass-stoppered bottle containing 10 cc. potassium iodide solution (10 per cent.) and 5 cc. dilute sulphuric acid, and agitated for several minutes; tenth-normal solution of iodine was then run in from a burette until the liquid in the bottle was distinctly brown in color after frequent agitation during ten minutes; the excess of iodine, finally, was titrated with tenth-normal solution of sodium thiosulphate. By subtracting the cc. of thiosulphate solution from the cc. of iodine solution and multiplying the difference in cc. by 0·00165 the quantity of hypophosphorous acid (free and combined) present in the weight taken will be obtained.

The tabular statement explains itself:

| Number of Sample. | Specific gravity at 25° C. compared with $H_2O$ at 25° C. | Hypophosphorous acid; grains per fluid ounce. | | Basic constituents present. $Q$ = Quinine. $S$ = Strychnine. |
|---|---|---|---|---|
| | | Found. | Calculated from formula. | |
| 1, . . . | 1·109 | 0·366 | — | K, Na, Ca, Fe, Mn, Q, S |
| 2, . . . | 1·299 | 2·232 | — | K, Ca, Fe, Mn, Q, S |
| 3, . . . | 1·284 | 1·615 | — | same |
| 4, . . . | 1·286 | 1·207 | — | same |
| 5, . . . | 1·291 | 26·175 | 26·520 | K, Na, Ca |
| 6, . . . | 1·285 | 24·313 | 24·764 | same |
| 7, . . . | 1·285 | 24·564 | 24·764 | same |
| 8, . . . | 1·278 | 24·710 | 24·764 | same |
| 9, . . . | 1·225 | 31·478 | at least | K, Na, Ca, Fe |
| 10, . . . | 1·225 | 31·071 | 29·709 | same |
| 11, . . . | 1·321 | 22·020 | 23·988 | K, Na, Ca, Fe, Mn, Q, S |
| 12, . . . | 1·337 | 7 695 | — | Na, Ca |
| 13, . . . | 1·308 | 12·618 | 14·210 | Na, Ca |
| 14, . . . | 1·293 | 2·893 | 3·518 | K, Ca, Fe, Mn, Q, S |
| 15, . . . | 1·290 | 11·792 | 10·584 | K, Na, Ca, Fe, Mn, Q, S |
| 16, . . . | 1·335 | 19·461 | 27·462 | K, Na, Ca |
| 17, . . . | 1·297 | 4·184 | 9·935 | Na, Fe, Mn, Q, S |

Some remarks upon these samples may prove interesting reading:

1 is a preparation extensively advertised as a "tasteless preparation of cod liver oil and the hypophosphites;" in appearance and properties it forms a splendid example of an elixir, the taste of which is not rendered unpleasant by homœopathic doses of hypophosphites, nor is the odor affected by substituting iodine for its equivalent of cod-liver oil.

2 is a "proprietary" and has been "made" such a success that numerous imitations are being offered to the trade; of these imitations 3 and 4 are examples. The samples so far (1–4 inclusive) probably owe more of their medicinal effect to the alkaloids than to the small quantity of hypophosphites 5, 6, 7 and 8 are samples of the official syrup of the hypophosphites; 5 containing free $H_3PO_2$ in place of citric acid, 6 strictly U. S. P.; both of these were made by me; 7 (recently made) and 8 (in stock for at least two years, during which time it had been repeatedly exposed in a partly filled bottle to direct sun-light) were obtained from a wholesale house; these samples show no difference in quantity of hypophosphite, although 8 was very much darker in color.

9 (old) and 10 (new) are Syrup of the Hypophosphites with Iron; they are not made according to the U. S. P. (which preparation speedily deposits insoluble ferric hypophosphite produced by the oxidation of ferrous hypophosphite when in contact with air and in presence of other hypophosphites; the ferrous hypophosphite formed by double decomposition of the ferrous lactate and the alkaline hypophosphites), but start with ferric hypophosphite and retain this in solution by the addition of some free hypophosphorous acid.

11, Syrup. Hypophosp. Comp. of the National Formulary; in this the precipitation of ferric hypophosphite is prevented by the presence of an alkaline citrate.

12 to 17, inclusive, illustrate the great army of special preparations placed upon the drug store shelves through the conversion of the practitioners by free samples, etc. As the results show, there are a number of these preparations, which excite suspicion that they never contained the quantity of salts which the labels state to be present.

It is hoped that the results obtained in the investigation can be used by pharmacists in convincing the physicians of their acquaint-

ance who are addicted to this weakness that there are preparations which can be made by the authority of the Pharmacopœia and National Formulary containing as much of the remedial agents in an ounce as some of their favored specialties contain in a pint.

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## THE CULTIVATION OF COFFEE IN JAMAICA.[1]

By C. G. Lloyd.

The island of Jamaica exports each year between eight and nine hundred thousand pounds of coffee, valued last year at a million three hundred and sixty thousand dollars, and the product was last year 15·7 per cent. of the total exports from the island. In former years the great bulk of this coffee went to England; thus only ten years ago, England got 73 per cent., while the United States only received 13 per cent.; but beginning with 1884 the States have taken a large proportion of the product, and last year received 45 per cent., the year before 57 per cent. I am very sorry to have to report, however, that the United States only gets the poorer grades, the English paying a better price for the choice grades. The best coffee of the island is raised on the Blue Mountains, in the parishes of St. Andrew and St. Thomas, the eastern end of the island, which coffee almost entirely goes to England. I am informed by the planters of Manchester parish, who sort their coffee, that their best grades, also, go to England.

Jamaica (and also Hayti) coffee is of an average good quality, a little stronger than Java or Mocha, but not so strong and rank as the Rio. A large New York importer of West Indies products told me that a certain coffee firm, whose name is a synonym for wealth, had made a fortune in the last half dozen years, selling roasted Jamaica and Hayti coffee as "choice Java." I presume every one who knows nothing of the subject, has an idea how coffee grows, even if it is erroneous. We naturally imagine that it grows on trees like cherries, and I had expected to see a coffee plantation look like a cherry orchard.[2] When I left Kingston by rail for the

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[1] Read before the American Pharmaceutical Association at the Profile House, N. H., July 16.

[2] My impressions had been formed from the picture plate 10, of the recent French work, "Plantes Médicinales," of Dujardin-Beaumetz and Egasse. This plate is so grossly inaccurate, not only in regard to the character and apparent

interior of the island, a couple of weeks before Christmas, having been told that the coffee berries were then ripe, I kept a sharp outlook for the coffee trees, but saw nothing that I could take for them. On arriving at the station, I walked along the single road or street of the little village of negro huts, and chancing to stop by the side of a copse of tangled bushes which I took for a wild growth, I noticed a few coffee berries on the ground under the bushes, and on investigating found that these bushes were coffee shrubs. I tried to think of what they reminded me at home, and nothing conveys to my mind a closer comparison than a tangled undergrowth of Wahoo shrubs.

The bulk of the coffee of Jamaica is raised by small growers—negroes, who own from a half to five acres of ground, and who plant the shrub around the place without any order or system whatever, and apparently give the shrub no attention, excepting to break off occasionally the tops when they get too high or to cut off a few dead branches. In the statistics of the island, where the estates are specified which raise fifty acres or more of coffee, only thirty estates are named, comprising about 3,000 acres, while the acreage of small holders, less than fifty acres, is nearly 18,000. These small growers, of course, for the most part have no machinery for preparing or sorting the coffee. Almost every negro hut in the coffee districts has in the yard what they call a "barbicue." It is a flat drying surface, built where the sun will strike it, and reminds one of a square tray on a large scale, built of brick with raised edges and cemented smooth. The usual size is from twelve to twenty feet square. The negroes gather the coffee berries when they get ripe, a few each week, somewhat like we would pick gooseberries, one at a time. They put the berries into a wooden mortar and beat them, which separates the outer skins, which are washed away in buckets of water. The seeds are then put on the "barbicue" to dry. Without a close examination at this stage the product resembles large grains of coffee mixed with the imperfectly separated outer skins, but on closer observation we notice that each grain of coffee is enclosed in a thick, tough, cartilaginous skin. When the coffee has

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size of the coffee tree, but also to the size, shape, color, and cluster of the berries, that it is a discredit to that otherwise very excellent work. A good illustration of a coffee branch is plate 106 of the German work just completed, "Köhler's Medicinal-Pflanzen Atlas."

been dried on the "barbicue" this skin becomes brittle, and the negroes again beat it in the mortar to hull it out of this skin. Then the seeds are picked over by hand, the better part of them being sold to the little stores throughout the country, which we notice with the sign out, "Licensed to deal in agricultural products," and which pay Her Majesty's government two pounds each per year for the privilege. These small storekeepers send it to Kingston, from whence it is shipped abroad. Coffee merchants in Kingston, and some of the merchants in the smaller towns, sort the coffee into grades according to size and weight of the berry. Most of the sorting is done by hand, though some have sizing machines, as described further on.

On coffee plantations the same process is gone through, but on a larger scale, more systematic, and with the aid of machinery. The coffee shrub thrives best on new land, hence the portion of the plantation devoted to coffee growth is virgin soil cleared of its forest for this purpose.

Around Mandeville, in Manchester parish, the land is now almost all pasturage, and I am told that the whole of it was originally cleared off for the growth of coffee many years ago in slave times, and having raised its crops of coffee and exhausted the ground for this purpose, it was sown in Guinea grass and used for grazing. To establish a coffee plantation the land is cleared of its trees, burnt over, and cleaned up. Then it is laid out by pegs into squares of six feet, and young coffee sprouts about a foot high are planted near each peg. These sprouts are generally obtained from beneath old shrubs, and are adventitious growths from seed dropped from the shrub, though sometimes nurseries are established for raising the young sprouts from planted seed. In these tropical regions, weeds and vines and wild growths of all kinds spring up very quickly, and with these the planter is constantly at war. Four times a year, at least, the fields should be gone over with a hoe and the weeds cut down. In three or four years the young coffee plants begin to bear, and the shrubs continue giving crops for about thirty years. The shrub, if left to grow, would reach a height of twelve to fifteen feet, but on a plantation they are topped when about four feet high, and kept to about this height by breaking off the tops and such suckers as appear. The branches are slender, and when the shrubs are not crowded, spread nearly horizontal. The leaves are evergreen (as

indeed are most of the shrubs and trees in the tropics), of a firm texture, smooth and shiny above. They are opposite, oval, entire, and borne on short petioles about one-half an inch long. They are three to five inches long, two to three inches wide, and are terminated by acuminate points.

The flowers are white, borne in clusters of three to six in the axils of the leaves, and are exceedingly fragrant. The petals are five, slender, spreading. The shrubs begin to blossom in February and continue in flower up to May; the fullest bloom is in March and April. Coffee does not blossom as our fruit trees, all at once, and go out of bloom in a week or two, but continues to bloom for about four months, and the crop in consequence ripens through the same length of time, and the planters are thus enabled to gather and care for it to better advantage than if it all ripened at once. The coffee season lasts from September to December, September and October being the principal months. The coffee berries are borne on short stalks in clusters of three to six in the axils of the leaves. When ripe they are about the size of cherries, but are oval (not globular), and slightly compressed on the side. Each berry consists of two seeds (familiar to us as the green coffee of commerce), each seed enclosed in a thick, tough white skin called the parchment skin, placed in the berry with the flat surfaces together, and surrounded with a small quantity of sweetened pulp, the whole enclosed in a thick skin like a Malaga grape. The color of the skin when ripe is red, not a bright red like a cherry, but a pale dull red.

The berries are picked by negro and coolie women, who go over the coffee shrubs, picking the ripe berries into baskets, and are paid by measure. The price varies according to abundance of the berries, but is regulated so that a woman makes about ninepence (18 cents) a day. Rats are very fond of the sweetish pulp that surrounds the coffee grains, and they climb the shrubs and gnaw off a great many berries. Birds are also said to pick them, and lizards—which are very numerous in Jamaica—are charged also with despoiling the fruit. This "rat" coffee is picked from the ground by the women, and comprises about one-fourth of the crop. It furnishes a larger proportion of heavy grains than the berries gathered from the shrubs, as the rats are credited with selecting the largest and best berries, and it is kept separate in all the subsequent operations. As the bulk of this coffee is supposed to be gnawed off by the rats, all

coffee picked up from the ground is called "rat coffee." It costs about double to gather it as when picked from the shrubs.

The women bring the berries to the works, where they are measured and paid for by the "Busher," as the overseer of a coffee plantation is called. To prepare the coffee for market the berries are first run through a machine, called the "pulper," which tears off the outer skins and pulp. A "pulper" is simply a large cylindrical wheel about three feet in diameter and two feet long, covered with corrugated iron, like a nutmeg grater, and arranged so that it revolves so close to another corrugated iron surface that the berries cannot go through entire, but are caught by the rough surfaces and torn to pieces, the skins and pulp being carried through, the seed dropping beneath into a tank of water. The water serves to wash the grains, and also to separate the light from the heavy coffee; the former floating, are skimmed off; the latter sinking, are taken from the tank after the water is drawn away. Heavy coffee is much the better grade, and it is kept separate from the light in all subsequent operations. At this stage the coffee seeds are still enclosed in the "parchment skins," which are tough and cannot be separated from the seeds when green; hence the next process is to thoroughly dry the seed in order to make the "parchment skins" brittle so they can be hulled off. For this purpose the seeds are spread on "barbicues" similar to those previously described, only, of course, on a larger scale. The "barbicues" of an ordinary sized plantation cover about an acre of ground, and are usually built on sloping ground and terraced. When it threatens a shower, and every evening to protect it from the rain and dews (which are heavy in the tropics), the coffee is raked into a pile in the center of each "barbicue" and covered with a wooden cover-shaped hopper. From ten days to two weeks' exposure to the sun on the "barbicue" will dry the seeds so that they can be hulled. The "huller" is a large wooden wheel, arranged to revolve like the wheel we see in brickyards, but running in a circular narrow trough. The coffee is placed in this trough, and the wheel constantly running over it breaks off the brittle "parchment skins," being heavy enough for this purpose, but not so heavy as to crush the seeds. The coffee seeds are separated from the broken "parchment skins," called trash at this stage, by being run through a "fanner," similar to the fans of our threshing machines, which blows off the trash. There still remain closely

adhering to many grains of coffee thin light gray skins, called "silver skins," which would hardly be noticed by the ordinary observer. To remove these skins and brighten the grains of coffee it is further dried in the warehouse for two or three weeks, and again put through the "huller" and "fanner." The next step is to separate the "pea berry coffee." A small percentage of the coffee berries, instead of containing the normal two seeds, have by abortion only a single seed. The grains of these single-seeded berries, instead of having a flat face, are rounded, and are called "pea berries." These "pea berries" are heavy and of the best quality, and bring a better price than the best grade of flat-faced grains. To separate them the coffee is run into a cloth belt slowly revolving at a slight inclined plane, the flat-faced grains being carried over the top, the rounded "pea berries" rolling off the bottom.

The coffee is next graded according to the size of the grains by being run through a "sizer." This is a cylindrical screen, consisting of four sections of different sized meshes, the smallest holes near the top. The screen revolves at an incline, and the different sized grains drop through the various sections according to size into bins beneath, the largest grained and best grade being carried through the cylinder.

Finally the coffee is given to women who spread it on a table and pick out all the deformed or broken grains, which are called the "tringe." The best grades of coffee are put in tierces holding about 800 pounds, and mostly shipped to England—the poorer grades and "tringe" into barrels or bags for this country.

I have given a description of the machinery which I saw in operation on the plantations. There are improved machines, I am told, but they are said to furnish no better results than the old ones.

In concluding this article, I wish to acknowledge my indebtedness for information and other courtesies to John H. Nosworty, the "Busher" of Somerset Plantation.

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## VERBENA URTICÆFOLIA.

By Robert M. McFarland, Ph.G.

Contribution from the Chemical Laboratory of the Philadelphia College of Pharmacy. No. 113.

The leaves of this plant have acquired some reputation in domestic practice, as a tonic; the usual mode of administration has been as

a decoction. The fresh root is also considered to be of some medicinal value, an infusion of the root having been administered with assured advantage in intermittent fevers. The taste of this infusion would lead one to suppose the medicinal activity to be due to a bitter principle.

In view of the above statements concerning this plant, it was thought that a chemical examination of the root might be of interest. For this purpose a quantity was collected by the author near Henderson, Kentucky, during the summer of 1891. It was carefully dried, and a proximate analysis made during the past winter, with the following results.

Fifty grams of the dried and finely powdered root were extracted with petroleum ether, which removed 0·91 per cent. of a substance consisting of a volatile oil, fat and caoutchouc. Stronger ether extracted from the remaining drug 0·55 per cent. of resin.

Absolute alcohol next extracted 2·74 per cent. This extract was found to possess the bitterness of the original drug. It was therefore dissolved in a small quantity of alcohol, and poured into acidulated water. The solution thus obtained was filtered from the precipitated resin, and found to hold the bitter principle. This aqueous solution was then agitated with petroleum ether, followed by an agitation with ether. Both of these solvents removed a crystalline substance, the character of which was not fully determined, but from experiments made with it appeared to be an acid. The aqueous solution was then agitated with chloroform, which extracted a substance with a bitterish and slightly nauseous taste. This amorphous principle was dissolved in water, and a portion tested for glucose with Fehling's solution, which gave a negative result. Another portion was then boiled for fifteen minutes with very dilute hydrochloric acid, neutralized and tested with Fehling's solution, which now gave abundant evidence of the presence of glucose. This principle, therefore, with a taste identical with the original drug, was undoubtedly a glucoside. The aqueous solution remaining from the agitation with chloroform had lost its bitter taste, and nothing further was removed by making it alkaline and again extracting with the above immiscible solvents, this indicating the absence of an alkaloid. The above glucoside was not further examined on account of the difficulty, at that season of the year, of obtaining a further supply of material from which to extract it.

It is suggested, however, that a method of preparation would be to extract the drug with alcohol, recover the solvent by distillation, and pour the residue into acidulated water. After standing a short time the mixture may be filtered to remove resin, and the acidulated aqueous solution agitated with ether to remove remaining resin and acid principle, and then with chloroform to extract the bitter glucoside.

The original fifty grams of drug, after treatment with absolute alcohol was extracted with successive portions of distilled water. This solvent removed 18·45 per cent., consisting of mucilage 2·40 per cent., dextrin 5·28 per cent., glucose 5·32 per cent., and saccharose 4·84 per cent. There were further found by appropriate solvents, pectin and albuminoids 3·84 per cent.; starch 1·76 per cent. The cellulose, lignin and incrusting matter amounted to 40·51 per cent. The ash was found to be 13·82 per cent., and the moisture 10·80 per cent.

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## ABSTRACTS FROM THE FRENCH JOURNALS.

TRANSLATED FOR THE AMERICAN JOURNAL OF PHARMACY.

*Oil of turpentine in lemon oil.*—Oliveri (*Gazetta chim. ital.*, xxi, 318, through *Rep. de Pharm.*, 1892, p. 221) examining oil of lemon for adulterations uses a polarimeter of Laurent with a 20 cm. tube. The rotatory power for pure lemon oil is + 120° and for turpentine — 55°. The rotatory power of mixtures is seen from the following table:

| | | | | |
|---|---|---|---|---|
| 2 | parts of | turpentine | in 100 | = 116·5° |
| 4 | " | " | " | = 113·00° |
| 6 | " | " | " | = 109·5° |
| 8 | " | " | " | = 106·° |
| 10 | " | " | " | = 102·5° |
| 15 | " | " | " | = 93·75° |
| 18 | " | " | " | = 88·50° |
| 20 | " | " | " | = 85·00° |

The examination is made at ordinary temperature 15–20°, variation having no effect on the observations.

*Use of the fruit of Sorbus Aria in Asia Minor.*—Duchesne (*Rep. de Pharm.*, 1892, 227) calls attention to these fruits which are about the size of a small nut and are known in Asia Minor under the name of idé. The inhabitants make use of the pulp of the fruit in the place of "farine lactée" in the feeding of infants, the pulp

being mixed with water or milk. Gautrelet analyzed the fruits, his results being as follows: The envelope and kernel make up about one-half of the fruit. The pulp contains glucose 11·44, sorbine 13·56, nitrogenous matter 6·85, cellulose 6·05, fat 0·50, carbonates, chlorides and phosphates of alkaline earths 3·41, water 8·00, the percentages being taken on the whole fruit.

*Action of the pancreatic liquid on the decomposition of salol.*—According to Nencki the antiseptic virtues of salol are due to the decomposition into phenol and salicylic acid. According to this author the pancreatic liquid plays the principal role in this reaction. Dr. Gley (*Soc. de biologie*, through *Rep. de Pharm.*, 1892, 230) experimented with dogs from which the pancreas had been taken. He found in the urine from such animals after exhibiting salol, salicylic acid. These results confirm the conclusions of Perrier and Patein who stated that the decomposition is due to alkalies which are found in the blood from wounds or in the intestinal track.

*Adulteration of iodoform gauze.*—In an article signed L. & D. (*Bull. commerc.*, 1892, 186) attention is called to a peculiar sophistication of iodoform gauze. The gauze examined was marked 30 per cent., but on analysis showed only 8 per cent.; the deficiency in color was made up with a nitro derivate of phenol. To look for this adulterant it suffices to treat the gauze with water when, if it be present, a yellow colored solution results, yielding on evaporation a golden yellow residue which, after being fused over charcoal, will not color ether and has lost its bitter taste. Properly prepared iodoform gauze should yield no coloring matter to water.

*Fumarine in a papaveraceous plant.*—Dr. Battandier (*Compt. rendus*, May 16, 1892) having studied for some time the extraction of glaucine from the leaves of *Glaucium luteum, L.*, endeavored to obtain the same alkaloid from *G. corniculatum, L., var. phœniceum.* He did not find glaucine, but papaverine. This latter alkaloid was characterized, (1) by the violet color produced by cold monohydrated sulphuric acid, which oxidizing agents change to brown, heat to grayish green, and which is destroyed by water; (2) by the chloroplatinate crystallizing in octahedra, and (3) by its behavior towards solvents. The alkaloids of *Hypecoum*, *Bocconia frutescens* and *Eschscholtzia californica* give with sulphuric acid a color similar to that of papaverine, but the chloroplatinates could not be crystallized.

Fumarine seems to exist in the genera and sub-genera Fumaria, Platycapnos, Sarcocapnos, Ceratocapnos, Corydalis and Diclytra.

*Resin oil in linseed oil.*—Dr. Coreil (*Journ. de Pharm. et de Chim.* Febr. 15, 1892) after reviewing the qualitative tests for resin oil in linseed oil proposes a quantitative test based on saponification with alcoholic potassium hydrate solution. The qualitative tests mentioned are, (1) the red and brown color produced by fuming bichloride of tin; (2) the non-saponification by alkalies; (3) odor and taste by which 5–10 per cent. of resin oil could be recognized; (4) the brown and black color produced by a current of chlorine. The quantitative test which, however, only comes near the truth, is as follows: 2 gm. of the oil are freed of air by heating to 105° C. for five or six hours, then heated on a water-bath with 40 cc. of a deminormal alcoholic solution of potassium hydrate, the remaining alkali being titrated with a deminormal solution of hydrochloric acid in the presence of phenolphthaleine. The number of cc. used to saturate the fatty acids of the oil is multiplied by 0·02805 and the product divided by 2 to obtain the quantity of potassium hydrate necessary to saponify 1 gm. of oil. The quantity of potassium hydrate used to saponify 1 gm. of linseed oil varies between 201–221 mg. (average 211), for resin oil 21–41 mg. (average 31). The final calculation is based on the fraction,

$$\frac{100\,(211 - n)}{211 - 31}$$

$n$ representing the quantity of potassium hydrate necessary to saponify 1 gm. of the oil.

*Blood oranges.*—A. Barillé, in the meeting of May 4th of the Société de Pharmacie de Paris (*Rep. de Pharm.*, 1892, 277), calls attention to a sophistication of blood oranges. The zest of the samples examined was artificially colored with Biebrich scarlet or rocceline, two azo colors which, however, are non-poisonous.

*Lead in commercial tartartic and citric acids.*—Dr. Buchet (*L'Union pharm.*, 1892, 203) found in one kilogramme of commercial tartaric acid from 0·011–0·071 gm. of metallic lead, and from 0·017–0·363 gm. of lead salts. The method of analysis used is as follows: 200 gm. of tartaric acid are dissolved in three times their weight of distilled water with a slight excess of ammonia to insure the solution of sulphate of lead. The solution is

then set aside for twenty-four hours, the liquid decanted and the residue collected on a small filter. The filter and contents are treated with nitric acid; to this solution sulphuric acid and twice its volume of alcohol are added. The precipitate of sulphate of lead is washed with alcohol, collected on a filter and weighed after calcination. This gives the quantity of metallic lead. To estimate the amount in combination the ammoniacal liquid from above is used. The liquid is slightly acidulated with hydrochloric acid and a current of hydrogen sulphide passed through it. It is then set aside for twelve hours, the sulphide collected and this dissolved in dilute nitric acid. The remainder of the operation is the same as above.

*Estimation of tannin.*—G. Fleury (*Journ. Phar. Chim.*, 1892, 499) proposes to use egg albumen for estimating tannin. The hard boiled egg albumen is dried at a moderate temperature and powdered. This is washed with dilute alcohol (10 per cent.), very slightly acidulated with tartaric acid to saturate the alkali. The albumen is again dried and kept in a well-stoppered bottle. The method of operation is as follows:

Albumen powder equal to seven or eight times the quantity of tannin which is supposed to be present is added to the liquid in a flat dish. The dish is then set aside for forty-eight hours, stirring occasionally; the liquid must during this time be acid and not alkaline. The end of the reaction is attained when the liquid does not give a color with perchloride of iron. The powder is then collected on a filter, is washed with a very dilute alcohol, and is then dried at 100° C. At the same time the amount of water in a sample of the albumen from the stock is determined, the difference between the two then giving the amount of tannin present. Gallotannic acid cannot be estimated according to this process, because the absorption by the albumen is incomplete and very slow. In testing it is necessary to bear in mind that gallic acid is not absorbed by the albumen and consequently still gives its reaction with ferric chloride.

*Dental and buccal antisepsis.*—Dr. Millon (*Monit. thérap.*, through *Journ. Pharm. Chim.*, 1892, 624) prescribes the following mouth-wash for the above purpose, a tablespoonful to be used with a tumbler of lukewarm water: Thymic acid (thymol), 0·25; benzoic acid, 3·00; tincture of eucalyptus, 15·0; alcohol, 100·00; oil of peppermint, 0·50 gm.

*Marine glue*, according to *Moniteur industriel* (*Nouv. Remèd.*, April, 8, '92, iv) is prepared as follows: About 450 gm. of caoutchouc are dissolved in 18 litres of benzol. When solution has taken place and the mixture assumed the consistency of a thick cream, which it usually does after about 10 days, shellac is added equal to two or three times the weight of the solution. The mixture is heated and run into plates. The glue is used at a temperature of 120° C.

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## GLEANINGS FROM THE GERMAN JOURNALS.

By Frank X. Moerk, Ph.G.

*Iodine-absorption of oils and fats.*—The method given in detail in the American Journal of Pharmacy, 1892, page 79, in which it was recommended to use four times as much iodine as was likely to be absorbed, has been modified, further research establishing that twice the amount of iodine was sufficient. The modified method answering for all fats and oils is as follows: About 0·150 gm. are dissolved in 10 cc. chloroform, 10 cc. each of iodine and mercuric chloride solutions are added and allowed to stand two hours; after adding 20 cc. potassium iodide solution and 100–150 cc. water, the excess of iodine is titrated with sodium thiosulphate solution. If in the determination it be found that the blank test requires less than twice the quantity of thiosulphate solution used in the case of the oil or fat, the determinations must be repeated, using 20 cc. each of iodine and mercuric chloride solution for 0·150 gm. oil or fat.—D. W. Fahrion, *Chemiker Ztg.*, 1892, 862.

*To determine alkalinity of Labarraque's Solution, etc.*—N. G. Blattner proposes several methods: (1) 25 cc. are diluted with 50–100 cc. water and after adding water of ammonia in excess the mixture is gradually heated until the reaction has ceased, when the liquid can be boiled to dryness and redissolved in water to make 250 cc. Of this solution, 100 cc. (= 10 cc. original preparation) can be directly titrated with standard sulphuric or hydrochloric acid, using methyl-orange as indicator. By using methyl-orange and phenolphthaleine it is possible to determine separately the alkalinity due to sodium hydrate and sodium carbonate. (2) 10 cc. of the preparation are diluted with water, a few drops phenolphthaleine added and titrated with normal sulphuric acid; it may be necessary to add a drop of phenolphthaleine solution from time to time to determine if the alkali has been neu-

tralized as the first portion may be decolorized through the liberation of small quantities of chlorine before the alkali has been neutralized. (3) 25 cc. are diluted with water and a little cobalt or nickel sesquioxide suspended in water added; after gradual heating (to prevent a violet reaction) the mixture is boiled, diluted with water to 250 cc., filtered, and in 100 cc. (10 cc. original solution) the sodium hydrate and carbonate are determined as under the first method. All three methods give satisfactory results; No 2 after some practice is to be preferred owing to rapidity.—*Chemiker Ztg.*, 1892, 885.

*Syrup of ferrous iodide*, according to A. Bernick, is a delicate reagent for ammonia, the color being changed to yellow or brown; this color is destroyed by boiling or by the addition of citric acid. It is thought that this is the explanation of the change in color that the syrup sometimes shows; at the same time the remedy suggests itself, either to boil such a syrup, or to add a little citric acid.—*Pharm. Ztg.*, 1892, 373.

*Constituents of insect powder.*—In continuing their investigations, Schlagdenhauffen and Reeb obtained, by distilling Dalmatian insect powder with steam, a pale yellow oil of a chamomile-like odor, in which is suspended a small quantity of a crystalline substance. The aqueous distillate contains formic, acetic and propionic acids and another organic acid, which was found to be poisonous, called *chrysanthemic acid*; the sodium salt of this acid is insoluble in alcohol. The residue from the distillation was found to still have toxic properties; by extracting this residue with petroleum ether, evaporating, dissolving in alcohol, neutralizing with potassa, evaporating to dryness, taking up with water, filtering, acidifying filtrate with tartaric acid, extracting with ether and evaporating the ethereal solution another poisonous acid, *pyrethrotoxic acid*, was obtained as a yellow, uncrystallizable, buttery mass, which was found very easily soluble in alcohol, chloroform, benzin, benzol, acetic ether and acetone. Caucasian insect powder yields almost identical results.—*Chem. Centr.-Blatt; Pharm. Ztg.*, 1892, 374.

*Iodoform deodorizer.*—Apparatus in which iodoform preparations have been made can be freed from every trace of the odor by the addition of a few drops of laurel oil before cleaning the apparatus with saw-dust. The odor of the laurel oil disappears in a very short time.—F. K., *Pharm. Ztg.*, 1892, 396.

*Russian peppermint oil* has been chemically examined by G. Andres and A. Andreef; they find that 17 volume per cent. of the oil is made up of the hydrocarbons lævo-limonene, menthene and pinene; the lowest-boiling fraction gave indications of a naphthylene $C_{10}H_{18}$. Experiments were also made proving that the distillation of the fresh and the air-dried herb gave identical products. The chief constituents of the oil are menthol and menthone.—*Berichte; Pharm. Ztg.*, 1892, 405.

*Agathine*, also called salicylaldehyde-*a*-methylphenylhydrazone, has been found to give good results in neuralgic and anti-rheumatic conditions in doses of 0·12–0·5 gm. (2–7 grains) two or three times daily: the action is not immediate but shows after a few days when 4–6 grams have been administered. The compound, when pure, forms small white laminæ melting at 72° C.; it is insoluble in water, but soluble in alcohol, ether, benzol, ligroin; boiling with concentrated hydrochloric acid decomposes it. The formula is given as $C_6H_4.OH.CH:N.N.CH_3.C_6H_5$; it is made by the Höchster Farbwerke.—(*D. Med. Ztg.*) *Pharm. Ztg.*, 1892, 414.

*Diamond cement.*—500·0 finest glue are allowed to soften and swell for several hours in 400·0 water and 100·0 acetic acid (96 per cent.); it is then warmed until dissolved and 1·0 pure carbolic acid added.

*Universal cement.*—250·0 sugar placed in a flask are dissolved in 750·0 water by aid of a water-bath, 65·0 slaked lime added and the mixture warmed for 3 days at 70–75° C., agitating repeatedly. After cooling, the supernatant liquid is poured off clear; 200·0 are diluted with 200·0 water and 550·0 finest glue allowed to swell in it for three hours, when it is heated until perfect solution takes place; after restoring the original weight by adding water, 50·0 acetic acid (96 per cent.) and 1·0 pure carbolic acid finish the preparation.

*Syndetikon.*—In 400·0 of the sugar-lime solution (see above) 600·0 ground glue are allowed to soften for three hours; after heating to effect solution and replacing the evaporated waste, the cement is neutralized with oxalic acid (about 30·0) and 1·0 carbolic acid added.—E. Dieterich, *Pharm. Ztg.*, 1892, 154.

*Cement for porcelain.*—20·0 white lead and 12·0 pipe clay, carefully dried, are incorporated with 10·0 boiled linseed oil heated on a water-bath; the cemented articles are dried slowly in a warm place. —(*Industrie-Bl.*) *Pharm. Centralhalle*, 1892, 330.

*Citric acid* may crystallize with or without water of crystallization; the anhydrous acid will crystallize again from cold aqueous solutions in the anhydrous form. By heating aqueous solutions of the hydrated acid to 130° C., the anhydrous acid is obtained; this melts at 153° C. If a crystal of the hydrated acid be placed in a cold saturated solution of the anhydrous acid, there will crystallize from the solution the hydrated acid, the reverse of this, however, has never been accomplished. It has been found that corresponding to the two modifications of the acid there can be prepared the corresponding lead salts. An error which has been introduced in many text books [U. S. P. also contains it—F. X. M.] gives the melting point of the hydrated acid at 100° C.; at 70–75° C. the hydrated citric acid sinters together, due to loss of water of crystallization; further heating produces little change until a temperature of 135–152 per cent. is attained, when it melts; the wide range 135–152° C. depends upon the rapidity of heating.—H. Witter, (*Berichte*) *Pharm. Centralhalle*, 1892, 353.

*Ash of kamala.*—In a sample recently received from J. H. Maiden which was collected in the northeastern portion of New South Wales, Professor Flückiger found only 3 35 per cent. ash. Climatic conditions apparently do not cause an increase in the amount of ash, as samples from northwestern India, central India, Java and New South Wales contained about three per cent. or less.—*Archiv der Pharm.*, 1892, 249.

## CONSTITUENTS OF ANGOSTURA BARK.[1]

By H. Beckurts and P. Nehring.

The bark of the Columbian rutacea, *Cusparia trifoliata*, Engler (*Galipea officinalis*, Hancock), has been examined by the authors.

30 kilos of the coarsely-powdered bark was extracted with 75 kilos of ether; most of the ether was then distilled off, and the brown residue extracted with dilute sulphuric acid. Wax and essential oils were thus separated from the alkaloids, and, in addition a greenish-yellow, crystalline substance was precipitated; it is the salt of a base or bases, but it could not be obtained pure.

From the acid solution, four alkaloids, *cusparine*, *cusparidine*, *galipine* and *galipidine* were separated. The separation of these

1 *Arch. Pharm.*, 229, 591–617; *Jour. Chem. Soc.*, 1892, 642.

was, however, exceptionally difficult, since their solubilities are very similar, and they were obtained pure only by means of (1) repeated fractional crystallization from their solutions in light petroleum; (2) fractional crystallization of the respective sulphates from hot aqueous solution.

The residual bark, after extraction with ether, was extracted with 60 kilos of alcohol, and the alkaloids, wax and oils separated from each other as from the ether extract. The same four alkaloids were again obtained. The total quantity of impure bases which was thus separated amounted to 595 grams, or 2·0 per cent.

*Galipine*, $C_{20}H_{21}NO_3$, crystallizes from light petroleum in slender, lustrous, soft needles which are pure white. It melts at 115·5°. It dissolves very readily in alcohol, chloroform, acetone, benzene and ether, but is only sparingly soluble in light petroleum. Its salts are yellow, and crystallize like the base. The *hydrochloride*, $C_{20}H_{21}NO_3,HCl,4H_2O$, forms lustrous, yellow plates, very soluble in warm water. The *hydrobromide*, $C_{20}H_{21}NO_3,HBr$, crystallizes in deep yellow needles, and is very soluble in water and alcohol. The *hydrogen sulphate*, $C_{20}H_{21}NO_3,H_2SO_4$, forms lustrous, yellow needles. The *platinochloride*, $(C_{10}H_{21}NO_3)_2,H_2PtCl_6$, melts at 174–175°, crystallizes in very small, yellow needles, and is almost insoluble in alcohol and in water. The *aurochloride*, $C_{20}H_{21}NO_3,HAuCl_4$, melts at 175–176°, and crystallizes in very small brownish-red needles. The *methiodide*, $C_{20}H_{21}NO_3MeI$, forms small, lemon-yellow needles which melt at 146°; it is soluble in alcohol and in hot water, insoluble in ether.

*Galipidine*, $C_{19}H_{19}NO_3$, crystallizes from light petroleum in very light, silky, lustrous plates which are pure white. It is readily soluble in alcohol, ether, benzene, ethyl acetate, and chloroform, less soluble in light petroleum; it melts at 111°. The salts are pale yellow, are readily soluble in hot water, and the solutions have a bitter taste. The *hydrochloride*, $C_{19}H_{19}NO_3,HCl + 3H_2O$, forms pale yellow needles, soluble in warm water, but only sparingly soluble in cold water; the *hydrobromide*, $C_{19}H_{19}NO_3,HBr$, crystallizes in minute, pale-yellow needles, readily soluble in water and alcohol; the *aurochloride*, $C_{19}H_{19}NO_3,HAuCl_4$, is precipitated as a mass of minute, chocolate-brown needles; it melts at 167°, and is almost insoluble in water and alcohol; the *platinochloride*, $(C_{19}H_{19}NO_3)_2,H_2PtCl_6$, separates as a voluminous, yellow, crystalline precipitate,

very sparingly soluble in water; it melts at 182°. The *methiodide*, $C_{19}H_{19}NO_3$,MeI, forms a fine, yellow, crystalline powder, readily soluble in hot water and alcohol; it melts at 142°, and has an intensely bitter taste.

*Cusparine*, $C_{20}H_{19}NO_3$ is comparatively easily separated from the other bases, owing to the sparing solubility of its salts. It melts at 89°, and is readily soluble in alcohol, ether, chloroform, acetone and benzene, more sparingly in light petroleum. Its salts are white and sparingly soluble in water, more readily in alcohol. The *hydrochloride*, $C_{20}H_{19}NO_3$,HCl + $3H_2O$, crystallizes in long, white needles, very slightly soluble in water, readily in alcohol, and of a bitter taste; the *hydrobromide*, $C_{20}H_{19}NO_3$,HBr, forms long, pale-yellow needles, of bitter taste; it is very sparingly soluble in water; the *sulphate*, $(C_{20}H_{19}NO_3)_2$,$H_2SO_4$ + $7H_2O$, crystallizes in white prisms of a bitter taste; it is only sparingly soluble in water, more readily in alcohol; the *aurochloride*, $C_{20}H_{19}NO_3$,$HAuCl_4$, forms a light-brown, voluminous mass of minute crystals, melts at 165°, and it is almost insoluble in water; the *platinochloride*, $(C_{20}H_{19}NO_3)_2$, $H_2PtCl_6$, forms minute, yellow needles, which may be recrystallized from hot water. It melts at 179°. The *methiodide*, $C_{20}H_{19}NO_3$MeI, forms lustrous, yellow needles, of intensely bitter taste, melting at 186°.

*Cusparidine*, $C_{19}H_{17}NO_3$, crystallizes from light petroleum in minute, slender, white needles. It melts at 78°, and dissolves readily in chloroform, alcohol, ether, and ethyl acetate, less readily in light petroleum. The salts are white and of bitter taste; they are less soluble than those of galipine and galipidine, but more readily than those of cusparine. The *hydrochloride*, $C_{19}H_{17}NO_3$,HCl + $3H_2O$, is precipitated as a white, voluminous mass of crystals, which melt very readily; it is sparingly soluble in water, more readily in alcohol, and has a bitter taste; the *hydrobromide*, $C_{19}H_{17}NO_3$,HBr, crystallizes from water in well-formed prisms, which are anhydrous; it is sparingly soluble in hot water, almost insoluble in cold water, more readily soluble in alcohol. The *sulphate*, $(C_{19}H_{17}NO_3)_2$,$H_2SO_4$ + $3H_2O$, separates as a crystalline precipitate when cusparidine is exactly neutralized with sulphuric acid; it is moderately soluble in water, still more readily in alcohol, and has a very bitter taste. The *aurochloride*, $C_{19}H_{17}NO_3$,$HAuCl_4$, separates as a voluminous, brown mass of minute crystals; it is anhydrous, and melts at 167°. The *platinochloride*, $(C_{19}H_{17}NO_3)_2$,$H_2PtCl_6$, forms a pale-yellow, crystal-

line precipitate, which is insoluble in water and alcohol; it melts at 182°. The *methiodide*, $C_{19}H_{17}NO_3MeI$, is prepared by heating cusparidine with excess of methyl iodide for three hours at 100° in a sealed tube; after recrystallizing from water, it forms a pale-yellow, crystalline powder, soluble in water and alcohol, insoluble in ether. It has a very bitter taste; its color changes to brown on exposure to the air or light, and it melts at 149°.

The four alkaloids are tertiary bases; their reactions with the various alkaloidal reagents are described.

The *essential oil* of angostura bark, which was extracted by means of ether, was freed from alkaloids by dilute sulphuric acid, and then remained as a dark-colored liquid. From 100 kilos of the bark, 1·5 kilos of oil was obtained. It has an aromatic odor; the sp. gr. at 15° is 0·956, and it dissolves in ether, alcohol, chloroform, light petroleum, and glacial acetic acid; it reddens litmus. It is free from sulphur and nitrogen, and does not react either as a phenol, a ketone, or an aldehyde. Distilled under a pressure of 35 mm., it commences to boil at 153°; the greater part distilled between 200° and 220°; the last portions became solid when cooled with a mixture of sodium sulphate and hydrochloric acid.

The *bitter principle angosturin*, is insoluble in ether, but soluble in alcohol, and is found therefore in the alcoholic extract of the bark; it is obtained free from alkaloids after the alcoholic extract has been rendered alkaline with sodium carbonate and extracted with ether. The bitter principle is difficult to obtain pure. The residue, after separating the alkaloids, was dissolved in water, and an excess of tannin added; the precipitate was washed with water, and dissolved in alcohol; lead acetate was added, and the precipitate first washed with alcohol and then with boiling water. The angosturin was further purified by dissolving it in water, precipitating with charcoal, extracting with alcohol and finally evaporating the latter. It remained as a transparent, pale-yellow mass, which is soluble in water, alcohol, and glacial acetic acid. The addition of ether to the solution in acetic acid precipitated the angosturin quite white; it melts at 58°.

The angostura bark contains a *glucoside*, which may be separated from the bark, after, exhausting it first with ether, and then with alcohol, by extraction with water. The solution fluoresces and reduces Fehling's and other metallic solutions. The glucoside was not, however, obtained pure.

# REACTIONS OF HYDRASTINE AND OTHER ALKALOIDS.[1]

By D. Vitali.

If a small crystal of hydrastine, or of one of its salts, is placed on a porcelain capsule and covered with concentrated sulphuric acid (0·5–1 cc.), it turns yellow, and, on stirring, the liquid acquires the same color; on adding a small fragment of nitre (an excess must be avoided), the color changes to a more or less intense brownish-yellow; if a solution of stannous chloride is now added drop by drop, the solution acquires a magnificent reddish violet color, the intensity of which depends on the amount of alkaloid present. This coloration is not destroyed on dilution with water.

If a particle of hydrastine is treated with nitric acid (4 to 6 drops), the alkaloid turns yellow, on heating for an instant to the boiling point, nitrous fumes are evolved, and, on evaporating to dryness at a gentle heat, a yellowish residue is left, which, when cold, is colored brownish-yellow by alcoholic potash, and remains as a greenish-brown mass on evaporating the alcohol. When cold, this becomes deep-violet on treatment with sulphuric acid. Solutions of hydrastine must be evaporated to dryness before applying the tests, which are sufficiently delicate to detect 0·0001 gram of the alkaloid.

*Bebeerine* turns blood-red on treatment as described with concentrated sulphuric acid and nitre, the color changing to green on the addition of stannous chloride.

*Codeine* turns dark brick-red when alcoholic potash is added to its solution after treatment with nitric acid, and coffee-colored when further treated with sulphuric acid; similarly, *narcotine* acquires an orange color on the addition of potash, the color changing to violet-red on adding sulphuric acid, and red to yellow on diluting with water.

A rather less delicate test for hydrastine is as follows: A particle of the solid alkaloid is fused with five or six times its weight of caustic potash, the melt allowed to cool, acidified with hydrochloric acid, extracted with chloroform, the extract evaporated to dryness on the water-bath, and the residue treated with a very dilute solution of ferric chloride; a fine, blue coloration is obtained if a few milligrams of the alkaloid has been employed; the color is destroyed by acids, and changed to brownish-red by alkalies.

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[1] *L'Orosi*, **14**, 405–416; *Jour. Chem. Soc.*, June, 1892, 755.

A characteristic reaction for *aconitine* is obtained by adding to it in small quantities a solution of potassium permanganate in sulphuric acid (1 : 200) and stirring; the green color of the reagent is replaced by a violet-tint, which disappears on further agitation, and is restored on adding more of the reagent, and so on. A point is ultimately reached at which the color is not affected by agitation, but at once disappears on diluting with water.

Clear indications of the presence of hydrastine in putrid animal matter cannot be obtained if the latter is treated by the Stas-Otto method, on account of the ptomaines and other impurities contained in the extract.

Hydrastine is, however, extracted from alkaline, but not from acid, solutions by light petroleum, and, by taking advantage of this fact, and substituting baryta for sodium carbonate, and light petroleum for ether, in the extraction, it is possible to isolate the alkaloid in a state of sufficient purity. The author recommends the use of light petroleum in place of chloroform, ether, or amyl alcohol in the extraction of alkaloids from urine and animal remains, as they are nearly all soluble in that menstruum (the exceptions are morphine, curarine, and pilocarpine) whilst ptomaines, leucomaines, pigments, and extractive matters are insoluble.

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## DIGITALIN.[1]

By H. Kiliani.

Hitherto the numerous attempts to obtain the pharmacologically important constituents of *Digitalis purpurea* in a state of chemical purity and in a crystalline condition have been without any practical result. Nativelle[2] believed that he had been successful in attaining that object, and for some time a preparation, made according to his directions, was used, especially in France; but it was afterwards given up, since it proved to be irregular in its action and indeed even hurtful. The "digitaline crystallise" of Nativelle was not a homogeneous substance, and in that respect it resembled all the other substances which have been introduced into commerce under the same or similar names. The recognition of this fact is

[1] *Archiv der Pharm.*, 230, p. 250; reprinted from *Phar. Jour. and Trans.* June 25, 1892, p. 1061.

[2] *Journ. de Pharm.*, iv, 9, 253; 16, 430; 20, 87.

most of all due to the meritorious investigations of Schmiedeberg,[1] the principal results of which are as follows:

The leaves and seed of *Digitalis purpurea* contain a preponderating amount of an inactive glucoside resembling saponin and named digitonin. They also contain three other substances which possess the characteristic power of acting upon the heart—crystallizable digitoxin and the two amorphous glucosides, digitalin and digitalein. Among these digitoxin does not appear to be suitable for therapeutic purposes, partly because great irregularity in its absorption is to be apprehended on account of its complete insolubility in water, and more especially because the investigations of Koppe[2] have shown that it also gives rise to very unpleasant and even dangerous effects. On the other hand digitalin and digitalein would be very suitable for practical use in medicine if it were possible to obtain them in a state of purity.

Several years ago I commenced a further investigation of the constituents of digitalis, and to some extent the results arrived at have been published.[3] Digitonin was obtained in crystalline condition, its composition was ascertained, the products of its transformation investigated, and it was further proved that the article then met with in commerce under the name of crystallized digitalin was nothing more than nearly pure digitonin, or in other words, a substance perfectly worthless in regard to action upon the heart, while in other respects directly injurious, as will be shown at the end of this communication.

I am now in a position to state that the solution of the main problem has been achieved, namely, the discovery of a practically applicable method of preparing the really active principle of digitalis in a state of purity. For this satisfactory result I am essentially indebted to the friendly assistance of Professor Boehm, of Leipzig, in carrying out the pharmacological testing of a large number of my products, by which I was led to the right path in my inquiries.

The conclusions thus arrived at were as follows:

(1) Besides digitonin and the active substances, the different kinds of commercial digitalin hitherto known generally contain two perfectly amorphous glucosides.

[1] *Arch. f. Exper. Pathol.*, 3, 16.
[2] *Arch. Exper. Pathol.*, 3, 274.
[3] *Ber. d. deutsch. Chem. Ges.*, xxiii, 1555; xxiv, 339, 3951.

(2) The digitalein of Schmiedeberg is certainly also a mixture; its action upon the heart is perhaps due to the presence of a glucoside that has not yet been separated in a state of purity, or perhaps it may be due to some admixture of digitalin.

(3) On the contrary, the digitalin of Schmiedeberg is a distinctly individual substance which possesses, in a marked degree, the characteristic property of acting upon the heart.

This substance is now being made according to my directions by Messrs. Boehringer, and it is to be obtained as a medicinal agent under the name of *Digitalin verum*. Unfortunately, this substance cannot be brought into an actually crystalline condition. For that reason it appears to be the more appropriate that its characters should be minutely defined, and that satisfactory evidence of its chemical individuality should be given.

Digitalin has the form of a white amorphous powder, the particles of which swell up when immersed in water. It is soluble in about one thousand parts of water, and in about one hundred parts of fifty per cent. alcohol. The water solution froths on being shaken, and it is remarkably prone to become mouldy. Digitalin is dissolved in considerable amount by hot absolute alcohol or spirit of 80 or 90 per cent., and when a minimum of solvent is used, the solution on cooling becomes almost solid from the separation of a thick magma of granules, which, as observed by Schmiedeberg, appears to the naked eye as if it were a case of crystallization. Examination by the aid of the microscope, however, shows that the mass consists of granules which, though of very uniform size, are entirely destitute of structure.

The uniformity of this deposit is, however, in itself a very characteristic feature of the substance; it is, moreover, an excellent criterion of its purity. When digitalin contains only a small percentage amount of the glucosides associated with it in digitalis, it is impossible to effect its separation from solution in the form above described. When it also contains some digitonin, and 85 per cent. alcohol is used for dissolving it, crystals will be found among the granules deposited from the solution after cooling.

These impurities of digitalin may be detected with greater accuracy by means of the following tests:

(1) A few granules when mixed with about 2 cc. of caustic potash solution (1 : 10) should retain their whiteness for at least one

minute. The presence of the minutest admixture of the amorphous glucosides is at once revealed by the rapid appearance of an intense yellow coloration.

(2) When digitalin is made into a thin paste with water, and there is added for every 100 parts of water, 22 parts amylic alcohol, on leaving the mixture for twenty-four hours in a closed flask, distinct crystalline warts will be apparent if digitonin be present even in very minute proportion.

Digitalin is almost insoluble in chloroform or ether; when the moderately dilute solution in absolute alcohol is gradually mixed with small quantities of ether the liquid suddenly becomes turbid, and within a short time there is a copious deposition of digitalin granules. On pouring off the solution and adding to it more ether a second point of saturation is reached, and with it a further deposition. The same effect may be produced a third time or more, and this behavior is evidence of the purity of the substance.

Contrary to previous statements the taste of digitalin is but slightly bitter; the intense bitterness and very disagreeable taste previously ascribed to digitalin are characters belonging to the amorphous glucosides with which it is associated in digitalis.

Digitalin dissolves in concentrated hydrochloric or sulphuric acid with a golden yellow color, and in the latter case the color rapidly passes into blood red. On adding to the solution, while still yellow, a drop of nitric acid, ferric chloride, or bromine water, a brilliant purple like that of digitalis flowers is produced, but the color is very evanescent. This coloration is obtained with more certainty, and so as to be permanent for one or two hours, when a very small quantity of digitalin is dissolved in ordinary sulphuric acid without any addition. Probably the small quantity of nitric acid commonly present in oil of vitriol is just sufficient to produce the coloration, but not sufficient for causing the further change which appears to take place by reason of the presence of an excess as compared with the digitalin when a drop of the oxidizing agents is added.

Digitalin remains white when heated to 200°; at 210° it begins to aggregate, and towards 217° it melts, becoming yellow-colored.

Analysis gave results agreeing with those obtained by Schmiedeberg which lead to the formula ($C_5H_8O_2$). This agreement may be taken as strong evidence that Schmiedeberg's digitalin was a chemic-

ally individual substance. However, the best support of this view is furnished by the behavior of digitalin with dilute hydrochloric acid. The substance is thus split up very definitely into digitaligenin, glucose and digitalose. When pure digitalin is used the first named product separates at once in fine crystals, but when the material operated upon contains some of the other glucosides, the digitaligenin cannot be made to crystallize at all, or only by very tedious operations.

In Schmiedeberg's experiments on the splitting up of digitalin with watery hydrochloric acid he obtained besides a glucose an insoluble resinous substance—digitaliresin—which he was able to decompose again into sugar and a second resin. On repeating this experiment it becomes at once obvious that this digitaliresin cannot possibly be a chemically individual substance, for it begins to be deposited from the first moment that heat is applied, and it may easily be seen that the resin carries down with it unaltered granules of digitalin. When the application of heat is continued for the purpose of decomposing the digitalin thus mechanically carried down, the acid causes a further decomposition at the surface of the resin, while inside the mass there still remains unaltered digitalin and digitaliresin. In order, therefore, to effect a definite splitting up, care must be taken that nothing is deposited while heating with the acid, and this may be done in the following way:

The digitalin is first mixed with 8 parts of 50 per cent. alcohol, 2 parts concentrated hydrochloric acid (1·19) added, shaken together, and heated for half-an-hour in a boiling water-bath under a reflux condenser. The liquid, clear at first rapidly becomes dark-colored, but does not during the heating deposit more than mere traces of resinous substance. Upon cooling, after removal from the water-bath, there is an abundant formation of brilliant acicular crystals of digitaligenin aggregated in warty masses. The crystallization takes place more readily on rubbing the sides of the glass, or on dropping in a crystal of the substance. On separating the crystals, washing with 50 per cent. alcohol, and drying, the quantity thus obtained amounts regularly to 30 per cent. of the digitalin operated upon. When the filtrate is mixed with an equal volume of water, repeatedly shaken with ether, and the ether shaken with very dilute soda solution to remove hydrochloric acid, a further quantity of the same substance is to be obtained. By repeated crystallization from the

least possible proportion of 93 per cent. alcohol, digitaligenin may be obtained in colorless crystals.

Digitaligenin is insoluble in water, sparingly soluble in ether free from alcohol, and readily soluble in alcohol, especially on heating. It dissolves in sulphuric acid or ordinary oil of vitriol with the same coloration as digitalin. The dry crystals of the pure substance become highly electric when rubbed. They melt at 210–212°. Analysis gave results leading to the formula $C_{16}H_{22}O_2$, and from the composition of several derivatives it appears very probable that this is a correct formula.

Professor Boehm informs me that when applied to frogs, digitaligenin has no action. For the purpose of identifying the remaining products of the transformation, the watery liquid which had been extracted with ether was again shaken with chloroform to remove a small quantity of resinous substance, then warmed to drive off chloroform and ether, and after cooling treated with silver oxide to separate hydrochloric acid. The liquid was then evaporated to a syrupy consistence, and left for some months to crystallize, partly alone and partly with addition of suitable solvents; but up to the present there have been no signs of crystallization, a circumstance which seems very remarkable when the further results of the investigation are considered.

An attempt was then made to obtain a knowledge of the nature of the sugar by preparing an osazone. Without much difficulty a product was obtained which crystallized very well, and was apparently an individual osazone melting at 206° C.; but the analysis of this substance obtained in different operations gave results which were but little in accord, and did not correspond with any known member of the class of compounds. Consequently, the presence of a mixture of sugars was to be suspected, and for their examination the method of oxidation by means of bromine proved very serviceable. From one of the two acids produced a lactone was obtained which crystallized well, and from the other a readily crystallizable barium salt.

The thick syrup was mixed with 2 parts of bromine after dilution with 5 parts of water, and by means of constant shaking the bromine was absorbed rapidly. After driving off the dissolved bromine and removing the hydrobromic acid by silver oxide, the liquid was evaporated to a syrupy consistence. Within some

hours crystals were formed abundantly (A); these were separated by a suction pump, and when washed with a few drops of water were nearly colorless. The mother liquor was diluted and boiled with barium carbonate, the filrate carefully saturated with alcohol, and left for some time in a closed flask, the sides of which were frequently rubbed. After some days warty crystals (B) were formed, which were separated from the mother liquor by washing with 50 per cent. alcohol and pressing. After separating the barium from the mother liquor by sulphuric acid and evaporating, a second crystallization of the substance A was obtained, and when the mother liquor from it was again converted into barium salt as before, a further quantity of the substance B was obtained. By this somewhat troublesome but certain method it was possible to separate the two acids produced by the action of bromine.

On recrystallizing the substance A, it was obtained in fine colorless crystals, readily soluble in water or alcohol, sparingly in ether; the crystals had a neutral reaction, and consisted of the lactone of a new acid, digitalonic acid. The air dry substance does not lose water in a vacuum over sulphuric acid; a little above 130° C. it begins to aggregate, and it melts at 138–139° C. Analysis gave results leading to the formula $C_7H_{12}O_5$. On heating with potash or soda solution, salts of the acid are obtained which do not crystallize. Moderately dilute solutions of these salts mixed with silver nitrate give in a short time an abundant deposit of silver digitalonate in microscopic needles, $C_7H_{13}O_6Ag$. When this salt is heated a product sublimes in fine needles.

From what has been stated it would appear that in the splitting up of digitalin the sugar produced, digitalose,[1] which has the composition $C_7H_{14}O_5$, and according to its behavior with bromine, it contains an aldehydradicle. In its composition it is closely related to rhamnose, and for that reason it ought to possess a great capability of crystallizing. Further investigation must determine what may be the explanation of the contrary character which has been described.

The barium salt B obtained from the mother liquor of the lactone was also readily purified by recrystallization; it then formed sharply defined laminæ having the appearance and composition of barium

[1] This name has been applied by Homolle and Quevenne (*Mem. sur la digitaline*) not to a chemically individual substance, but to a mixture of glucosides and their product of decomposition.

gluconate $(C_6H_{11}O_7)_2Ba + 3H_2O$.[1] A calcium salt prepared from it, corresponded in all respects with ordinary calcium gluconate. The lactone prepared according to the directions of E. Fischer[2] from the calcium salts gave the dextro rotation corresponding to d-gluconic acid, and hence it may be inferred that in the transformation of digitalin grape sugar is produced.

On the basis of the assumption, already referred to as being probably correct, that the composition of digitaligenin is represented by the formula $C_{16}H_{22}O_2$, the following equation is obtained: $C_{16}H_{22}O_2 + C_6H_{12}O_6 + C_7H_{14}O_5 - H_2O = C_{29}H_{46}O_{12}$, and a formula for digitalin $C_{29}H_{46}O_{12}$ which differs only by $CH_2$ from $C_{30}N_{48}O_{12} = 6 \times C_5H_8O_2$ or that indicated by the results of analysis of the glucoside. Whether that is really the correct expression of the molecular weight of the substance must be determined by other experiments.

However, it is sufficiently evident from the facts above stated that digitalin is to be regarded as having chemical individuality, notwithstanding the want of capability to crystallize. It follows that its physiological action must be constant, as distinguished from the varieties of commercial digitalin, hitherto known, which have been mixtures of the active and inactive constituents of digitalis, in variable proportions which rendered any safe adjustment of dose impracticable.

In the pharmacological trial of digitalin Professor Boehm has made the following observations:

Digitalin administered to frogs in doses of 0·5 milligramme produces systolic stoppage of the heart after 15 to 20 minutes. Increase in the volume of the heart pulse could not be detected by means of the Williams Dresser apparatus, even when the doses were very carefully administered.

Intravenal injection of 2 milligrammes produced in dogs increase of blood pressure with reduction of the frequency of the pulse and increase of its volume. By increasing the dose to 4 milligrammes

[1] The air-dry barium gluconate should theoretically lose 9·29 per cent. in drying, but the salt obtained gave only 8·17, and this was found to be the case also with barium gluconate (8·07). Herzfeld (*Annalen*, 220, 335) obtained similar results by heating barium gluconate to 115° C. By an error of calculation he gives as the theoretical amount of water 8·13 per cent.

[2] *Berichte*, xxiii, 2615.

arhythmia was produced, and after a short time sudden stoppage of the heart.

The same effects were produced on the cat by 1 and 2 milligrammes, respectively.

Rabbits were found to be less sensitive. Subcutaneous injections of .5 to 8 milligrammes, respectively, produced no effect. By intravenal injection of doses up to 8 milligrammes neither the blood pressure nor the pulse frequency was sensibly affected. Gradual increase of dose to 15 milligrammes caused after some time sudden stoppage of the heart even in rabbits.

The first trials of the action of digitalin on the human subject were made by Dr. Mottes in Munich, and he obtained the best results with doses of 0·25 milligramme at intervals of from 2 to 3 hours, without the occurrence of any disagreeable or dangerous symptoms.[1]

The use of pure digitalin in medical practice appears to recommend itself, not only in regard to the possibility of securing certainty in the dose. Hitherto it has been found that after long administration of digitalis preparations, hurtful and so-called cumulative effects have been observed, manifesting themselves especially in derangement of the stomach. This secondary action is in all probability to be ascribed not to digitalin itself, but to those substances which are associated with it in the digitalis preparations which have hitherto been used.

In any case this inference is warranted by the results which Professor Boehm has obtained in the investigation of digitonin. That substance, unfortunately, is the only one naturally associated with digitalin, except digitoxin, which has been up to the present time prepared in a state of absolute purity. It is also the one which occurs in the largest proportion. In regard to its action Professor Boehm reports as follows:

Digitonin exerts a very energetic action in the production of local inflammatory symptoms.

Administered internally to a dog in doses of 0 1 to 1·0 gramme, it produces in a short time vomiting, by which, of course, any further action upon the stomach or intestine is prevented. When given in doses of less than 0·1 gramme for ten days, it did not

[1] Professor von Ziemssen has also tried digitalin in the Munich hospital and obtained very good results.

appear to have any effect. But when injected subcutaneously, the dog refused to eat for two days, was very irritable, and would not suffer the injected places to be touched. At the end of the third day large pieces of skin were detached from the places injected, as if cut out with a knife down to the muscles of the back, and abscesses were formed, which rapidly healed.

In frogs, also, digitonin produces great local inflammation, in consequence of which these more sensitive animals rapidly succumb. In one instance marked nervous affection was noticed, with violent convulsions.[1]

For the purpose of ascertaining whether pure digitalin would produce local irritation when administered in the dose requisite for therapeutic purposes, Professor Boehm instituted experiments with the same healthy dog which had suffered inflammatory effects from the injection of digitonin. The animal was subcutaneously injected with 3 milligrammes of digitalin, dissolved in 1 cc. of water, with a few drops of spirit. The dose was strong enough to produce a powerful effect, which lasted for nearly 24 hours; but at the place of injection there was not the slightest inflammation or other change to be detected. It may, therefore, be concluded that true digitalin does not produce inflammation when administered in the dose requisite for exerting its full general action, and, therefore, trial of subcutaneous injections will be admissible in the human subject.

Moreover, this experience justifies the assumption that the substances associated with digitalin and present in the preparations hitherto met with in commerce, as well as in the infusion and digitalis powder, contribute to the production of the so-called cumulative effects.

It must be admitted that digitalin has one essentially dark side in its want of crystallizability, and this I have endeavored in various ways to find a remedy for. The chief obstacle encountered was the fact that even slight chemical alteration of the substance completely destroys its activity. Thus, for instance, when digitalin is heated only for a few minutes with acetic anhydride a finely crystallizable product is obtained, the analysis of which shows that it has the

[1] This observation justifies the desire already expressed, that the name of "crystalline digitalin" should no longer be applied to a preparation consisting almost entirely of digitonin.—See *Berichte*, xxiv, 3953.

formula $C_{29}H_{44}O_{11}$, or that of a hydride of digitalin. Professor Boehm has made trial of it and found that it is entirely inert.

*Note on the Preparation of Digitogenin.*—The transformation of digitonin into digitogenin, dextrose, and galactose, by treatment with hydrochloric acid[1] takes place as already pointed out[2] in a comparatively definite manner. It is, however, subject to the disadvantage, especially when operating upon a large quantity, that the digitogenin separates in an amorphous condition, and is, therefore, very difficult to wash out and dry before it can be brought into a crystalline condition by means of chloroform or alcohol. There is also a possibility that the separated digitogenin may be contaminated either with portions of digitonin or with the first product of its transformation, digitoresin, either of which would thus escape complete decomposition.

The favorable results obtained in the transformation of digitalin in an alcoholic solution, therefore, induced me to make a trial of the same method with digitonin. The result was very satisfactory; but to ensure success strong alcohol must be used, and the heating must be continued for a long time. Experiments made by Sanda showed that the following is the best method of operating:

A mixture of digitonin ($C_{27}H_{46}O_{14} + 5H_2O$) with 8 parts of 93 per cent. alcohol, and 2 parts concentrated hydrochloric acid (1·19) is heated for an hour and a half in a boiling water-bath under a reflux condenser. The whole is then allowed to cool slowly, and gradually the whole liquid becomes filled with warty masses of digitogenin. This deposit is separated by a suction pump, washed with 80 per cent. alcohol and pressed dry. It amounts to from 23 to 27 per cent. of the glucoside operated upon. For conversion into digitogenic acid it requires no further purification. A further quantity may be obtained from the filtrate from this first crystallization. For that purpose the liquid is mixed with calcium carbonate until effervescence ceases, the greater part of the alcohol is then distilled off, the residue diluted with water and shaken out with chloroform. The chloroform solution is freed from water by sodium sulphate, chloroform distilled off, and the residue of impure digitogenin is crystallized from 93 per cent. alcohol. About 5 per

[1] *Berichte*, xxiii, 1555.

[2] *Berichte*, xxiv, 3951.

cent. more digitogenin is thus obtained, making the entire yield about 30 per cent., while theory requires 36·8 per cent. This difference is to be accounted for by the circumstance that the alcoholic mother liquors from the recrystallization of the impure digitogenin obtained from the chloroform solution contain also a resinous substance—Schmiedeberg's digitoresin—which would yield an additional quantity of digitogenin by further treatment with alcoholic hydrochloric acid, the transformation not having been completed in the first operation. It is interesting to note the fact that in this second transformation it is chiefly galactose that is produced. The dextrose appears to be more readily eliminated, and, therefore, to be separated first.

## IMMUNITY AND CURE.[1]

By Dr. Klemperer.

Behring and Kitasato were the first to prove that, in animals, diphtheria and tetanus could be cured by treatment with the blood serum of animals which had been rendered insusceptible to these two diseases. By later authors the same remarkable fact has been proved with regard to swine-erysipelas, and the disorder caused by the bacillus pyocyaneus.

Klemperer has succeeded in adding two more diseases to the list, namely, mouse septicæmia (described by Koch in 1878) and the pneumonia caused by Friedländer's bacillus.

White mice are exceedingly susceptible to the bacilli of mouse-septicæmia, whilst rabbits are much less susceptible. Mice, after the injection of the minutest quantity of the bouillon culture, die to a certainty in from two to three days. Rabbits, on the contrary, can stand a good few cubic centimetres of the fluid injected subcutaneously, and even more when the injection is intravenous, without showing any evident sign of disease. Still, in spite of this natural immunity, the blood serum of rabbits proved powerless to cure, or even clearly delay the progress of the disease in mice.

The rabbit, however, is not completely insusceptible to the bacilli of mouse-septicæmia. Inoculate a rabbit with the virus in the ear; an erysipelas-like inflammation extends over the ear to its root, and there stops, and the animal usually recovers. It is now insusceptible to further inoculation, the immunity holding good while the inocula-

[1] *Berl. klin. Woch.*, March 28; *The Medical Chronicle*, July, 1892.

tion is subcutaneous or intravenous. With the blood serum of these rabbits thus rendered insusceptible, Klemperer succeeded in curing septicæmia in mice.

In mice the course of the disease (septicæmia Koch) is characteristic. A mouse has an injection of 0·1 ccm. of the fresh bouillon-culture. Within 24 hours there are, as a rule, no signs of illness, but on the morning of the second day there is a great change. The creature sits cowered in a heap with its eyes plastered to. As the disease progresses it curls itself up more and more, and finally dies in this position in the course of the third day. By injecting 0·5–1·0 ccm. of the blood serum of a rabbit rendered insusceptible, Klemperer managed to avert the disease altogether, and this was the case whether the injection of the serum was 24 hours in advance or injected at the same time as the bacillus culture. But—and this is of more importance—a cure resulted in every case when the blood serum was introduced 24 hours after infection, and in three cases 48 hours after infection. In these last three cases the mice had already assumed the characteristic posture, and the control mice died in a few hours. The affected recovered but slowly after the serum injection, and it was some time before they became lively and lost their curved attitude.

The efficacy of this serum was tried on other germ diseases, but in every case the result was negative. It was found also that mice cured by the serum were proof against later infection by mouse septicæmia bacilli. How long the period of immunity may last Klemperer cannot, as yet, say. Injections on the eighth and fourteenth day were negative.

Klemperer repeated a similar set of experiments with Friedländer's pneumonia bacillus with precisely similar results.

He also experimented with the bacillus pyocyaneus, and with similar success. Here, also, as in the two other diseases, he found that the blood serum of an animal rendered insusceptible could *cure that one disease only*, being absolutely useless when employed against any other germ.

These are Klemperer's facts—we now come to his conclusions. In the wonderful *transmission of immunity* lies the kernel of the matter.

The blood serum does *not destroy* the bacillus, either the mouse septicæmia bacillus or Friedländer's. A few bacteria in 5 ccm.

serum developed freely, and when, after twenty-four to forty-eight hours, they were transplanted on to bouillon, they throve and proved virulent.

When an animal recovers without artificial aid, this means that at length it has become insusceptible. The cure has resulted not from the dying out of the virus, but from the animal's becoming insusceptible to its action. On this theory the introduction of the immunized serum only hastens the process. The serum, however, of animals naturally insusceptible is powerless to avert or cure. There is, therefore, a difference between natural and artificial immunization. Still this difference may be only one of *degree*. Natural immunity is only relative. Immunity altogether is only relative, the natural being weak and the artificial strong, but still not absolute. The difference between the natural blood serum of an insusceptible animal and immunized serum may be only quantitative, as Kruse and Pansini have found that once when they injected 0·5 ccm. of the blood serum of a dog (naturally insusceptible to the pneumococcus) into two mice twenty-four hours before inoculation with the pneumococcus, they succeeded in producing immunity.

[These experiments are startling. When we have discovered what changes have taken place in immunized serum, wherein it differs, chemically or otherwise, from its former composition, then we may be able to cure disease witl out using the disease itself to produce its antidote. We seem to be on the brink of great discoveries.—W. A. STEWART.]

## PROTEIDS OF MAIZE.[1]

BY R. H CHITTENDEN AND T. B. OSBORNE.

The proteid matter soluble in 10 per cent. aqueous sodium chloride, but insoluble in water, may be resolved by fractional heat coagulation into three distinct globulins, two of which have much in common with phytomyosin and phytovitellin, although they are not identical with those substances.

The globulin which gradually coagulates when the temperature of a sodium chloride solution of the proteids, prepared in the manner previously described, is slowly raised to 80°, contains rela-

[1] *Amer. Chem. J.*, **13**, 529–552, and **14**, 20–40; reprinted from *Jour. Chem. Soc.*, June, 1892, p. 746.

tively less nitrogen, the average percentage composition of several specimens made in this and other ways being: C, 52·70; H, 7·02; N, 16·74; S, 1·30; the nitrogen of the undifferentiated proteid being 17·82 per cent. The globulin thus approximates closely in composition to animal myosin, which contains per cent.: C, 52·82; H, 7·11; N, 16·77; S, 1·27; but the bulk of it coagulates at 70°, a temperature markedly different from the coagulation point of the myosin. The globulin also resembles the vegetable myosin which was obtained by Martin from papaw juice (1886), and may thus be called *maize-myosin*. The tendency to coagulate at several different temperatures may be explained by assuming that maize-myosin undergoes a gradual change when heated, and this assumption accords with Martin's observation (1887), that the myosins of wheat and rye are transformed into insoluble substances by heating their sodium chloride solutions at 35–40° for some time, and with Vines' view (*J. Physiol.*, **3**, 91–114) that phytovitellin may be transformed into phytomyosin. Maize-myosin is characterized by its solubility in cold, dilute, saline solutions, such, for instance, as those of the alkaline phosphates naturally present in maize meal, and by taking advantage of this property may be extracted directly from the meal without dissolving the second globulin. The cold aqueous extract of the meal is saturated with ammonium sulphate, and the crude maize-myosin thus precipitated is successively treated with water and 10 per cent. aqueous sodium chloride. The mixed extracts are then dialysed in a stream of water, and the myosin thus precipitated is washed with water, alcohol, and ether, and dried.

The second globulin, *maize-vitellin*, which forms the bulk of the original proteid matter, is obtained in small spheroids when the solution from which the maize-myosin has been separated by heat coagulation is dialysed in a stream of water. Its solution in aqueous sodium chloride is coagulable by acetic acid, but not by heat, and it contains more nitrogen than the original proteid matter, the average composition of several specimens prepared in this and other ways being: C, 51·71; H, 6·83; N, 18·12; S, 0·86; thus closely approximating to that of the phytovitellin of pumpkin seeds: C, 51·88; H, 7·51; N, 18·08; S, 0·60. It contains less sulphur than the myosin, and is, perhaps, as much akin to heteroalbumose as to the true globulins. Maize-vitellin is distinguished from maize, myosin by its solubility in weak, cold, saline solutions, and by its

tendency to separate in spheroidal form from sodium chloride solutions. It may, therefore, be extracted without the aid of heat coagulation from the crude proteid matter, prepared in the manner previously described, or from the meal remaining after the extraction of the myosin. The proteid matter, for example, is dissolved in 5 per cent. aqueous sodium chloride, and the vitellin is first precipitated by dilution with a large bulk of water, then redissolved by heating the whole to 45°, and finally caused to separate in spheroidal form by cooling the solution slowly to 8°. The crude product thus obtained may be purified and caused to aggregate into larger spheroids by repeating the treatment. Maize meal from which the myosin has been previously extracted by cold water may be treated with 10 per cent. aqueous sodium chloride, the crude vitellin precipitated by saturating the solution with ammonium sulphate, and purified by a somewhat long process which comprises dissolving the crude product in sodium chloride solution, reprecipitating it by dialysis, dissolving it again in salt solution, heating the solution to separate coagulable impurities, precipitating the now nearly pure product with acetic acid, redissolving the precipitate in sodium chloride solution, and, finally precipitating the pure vitellin by dialysis.

The third globulin, which is characterized by its solubility in dilute solutions of salts other than chlorides, and its insolubility in water, is obtained by long-continued dialysis of the solutions from which the other globulins have already separated. It was not observed in the preliminary experiments, owing to the slow rate at which ammonium sulphate and the alkaline phosphates diffuse. This globulin has the composition: C, 52·38; H, 6·82; N, 15·21; S, 1·26, and is thus distinct from the myosin and vitellin. It is further distinguished from these globulins by the low temperature, 62°, at which it is completely coagulated.

Various other substances isolated during the investigation were probably formed from the solutions by the incidental treatment, and do not exist as such in maize.

A substance, for instance, soluble in water, not coagulated by heat, and in general behavior resembling the proteoses, was left in solution in the dialyser after the removal of the myosin and vitellin in the differentiation experiments first described. It did not exist in the original proteid substance, since the latter did not yield

any proteid matter to boiling water; possibly it was formed by the hydrolysis of one of the less stable globulins.

Another substance, precipitated by the addition of a slight excess of very dilute hydrochloric acid to the sodium chloride solution of the undifferentiated proteid matter, was possibly an acid globulin. It contained 17·39 per cent. of nitrogen.

The most prominent of these secondary products, however, are certain substances insoluble in aqueous sodium chloride, which are invariably formed during the dialysing operations by the prolonged action of the water or sodium chloride on the myosin or vitellin, which in this respect resemble animal myosin and certain vegetable globulins. When, for instance, the clear sodium chloride extract of maize meal is dialysed, the precipitated globulins are no longer completely soluble in sodium chloride solution; and the same thing happens when the globulins are precipitated by the addition of ammonium sulphate to the solution, especially if the sulphate is somewhat acid, although in the latter case the insoluble portion of the precipitate may contain the acid globulin mentioned above. In the early stages of transformation, these insoluble substances resemble the alkali albuminates, for they dissolve in dilute aqueous sodium carbonate, and are reprecipitated on neutralizing the solution; but after more prolonged action they seem rather to resemble coagulated proteid matters. Their composition is fairly uniform; the mean percentage composition, for instance, of the products formed by the action of aqueous ammonium sulphate or water on the sodium chloride or aqueous extracts of maize meal and subsequent purification was: C, 53·45; H, 6·99; N, 16·11; S, 1·14; so that if these substances are formed from the globulin, the latter must undergo considerable modification. The change is not due to the alkali used in the purification, since a specimen which had not been purified in this manner was found to contain the same percentage of nitrogen. Since this percentage is low, it is probable that the insoluble substances are formed from the myosin and the soluble globulin, and not from the vitellin, and it is probably this tendency of the first two to pass into such insoluble modifications which facilitates the purification of the vitellin.

An aqueous or sodium chloride extract of maize meal yields, after the three globulins and the soluble salts have been removed by dialysis, two albumins and a proteid soluble in alcohol (proteose). The solu-

tion is treated with 10 per cent. of sodium chloride, and rendered slightly acid with 0·2 per cent. hydrochloric acid. The precipitated albumin (I) dissolves when washed with water, doubtless owing to the presence of hydrochloric acid; it is reprecipitated when the solution is carefully neutralized with dilute sodium carbonate, but dissolves in excess of the latter; after drying, this precipitate is no longer soluble in sodium carbonate.

The filtrate from (I), when boiled, yields a coagulum of a second albumin (II); the portion first formed contains appreciably less nitrogen than the later portions.

The filtrate from (II) contains a proteose (III) which may be obtained by adding absolute alcohol to the concentrated solution. It is doubtful if these three substances all exist as such in the maize kernel; some of them may be formed in the processes used to separate them.

The maize meal, after extraction with water, contains another proteid, *zein*, or *maize-fibrin* (IV), which can be extracted with warm 75 per cent. alcohol, and is precipitated when the alcoholic solution is poured into water. It is quite insoluble in 0·5 per cent. sodium carbonate, and in 0·2 per cent. hydrochloric acid; it dissolves in 0·2 per cent. aqueous potash, but is not converted into alkali-albumin, for the precipitate produced when the alkaline solution is neutralized is not soluble in excess of dilute acid, but dissolves in alcohol. It is converted, when warmed with water or dilute alcohol, into an insoluble variety, which, however, has the same chemical composition. Both of these varieties exhibit the ordinary proteid reactions. The carbon-content of this proteid is large.

The results of analyses of these substances are tabulated below:

| | I. | II. | III. | IV. |
|---|---|---|---|---|
| C, . . . . | 52·86—53·53 | 51·02—52·06 | 50·07—51·13 | 54·97—55·42 |
| H, . . . | 6·86— 6·79 | 6·57— 6·79 | 6·54—6·91 | 7·15— 7·35 |
| N, . . . | 15·69—15·41 | 17·28—15·78 | 15.88—16·59 | 16·01—16·31 |
| S, . . . | 1·48 | 25·75—24·64 | 2·37— 1·62 | 0·53— 0·67 |
| O, . . . | 23·26 | | 24·52—23·75 | 21·05—20·52 |

# ON MAIZE OIL.[1]

By J. Cruickshank Smith, B.Sc.

The grain of the maize plant (*Zea Mais*), which is extensively used in the manufacture of starch, can be made to furnish an oil, whose properties would seem to render it suitable for several technical applications. At the suggestion of Professor E. J. Mills, the author recently examined a sample of this oil which is believed to be genuine, and the results are embodied in the present paper. The sample was of a bright golden color; it had a somewhat peculiarly "starchy" odor, and its taste was mild and not unpleasant.

Its specific gravity at 10° C. was 0·9267; at 15° C., 0·9244; and at 20° C., 0·9218. The mean co-efficient of expansion (corrected for glass) between 10° and 20° C. was 0·000706. Schaedler (Technologie der Fette und Oele) gives the specific gravity at 15° as 0·9215, and Professor Mills states that in 1884 he found a rather coarse brown sample to have a specific gravity of 0·9262. At — 10°, according to Schaedler, the oil sets to a solid mass. The author finds, however, that when exposed to intense cold, the oil becomes turbid, owing to the decomposition of solid matter at — 10°, and that below that temperature it becomes very viscous, but is still fluid at — 20°. The precipitated solid matter is entirely re-dissolved on warming again.

The bromine and iodine absorptions were as follows:

| | Per Cent. |
|---|---|
| Bromine absorption, | 66·50 |
| Iodine absorption, | 122·90 |
| Iodine absorption (calculated; Br. absorption $\times \frac{1.2.7}{8.0}$) | 105·50 |

It was observed that when the bromine solution remained in contact with the oil for more than fifteen minutes the results obtained were somewhat higher, and did not agree among themselves, owing, no doubt, to a secondary oxidizing action. A thick dark-colored sample was found by Professor Mills in 1881 to have a bromine absorption of 74·42 per cent.

On saponification with alcoholic potash, the total KOH absorbed was 19·34 per cent., which gives a "saponification equivalent" of 290·07. The oil was readily saponified in the cold, and it was at first thought that this ready saponification might be made use of for

[1] Abstract from the *Journal of the Society of Chemical Industry*, June 30, 1892; reprinted from *Phar. Jour. and Trans.*, July 16, p. 47.

the quantitative separation of maize oil from other oils; but while no other oil that has been examined gives such a high absorption in the cold as maize oil, many of them are undoubtedly acted upon under similar conditions, and concordant results could not be obtained.

The higher fatty acids present in maize oil seem to present no unusual features, but exact determinations as regards the proportion in which they are severally present were not made in the present instance. The volatile fatty acids, separated by Reichert's distillation process, in 100 parts of the oil required for their neutralization 0·56 parts of KOH.

A tendency to oxidize or to gum is almost absent in this oil. No decided siccative properties are communicated to it by simply "boiling" or by the addition of litharge to it. On passing a current of air through it for an hour at a temperature of about 150° C., the oil becomes slightly darker in color, and rather more viscous, but by no means to the same extent as cotton oil. If to the oil so treated a small quantity of borate of manganese is added, the oil acquires to a small extent siccative properties, and a thin film on lead dries in from ten to twenty hours, but not completely, being tacky to the feel at the end of that time. Like cotton-seed oil the eläidin reaction gives rise to a mass having a pasty or buttery consistency.

The rise in temperature when 5 cc. of strong sulphuric acid were mixed with 15 grm. of the oil was 89·0° C. For a non-drying oil this is a high figure, but it is in keeping with what may be called the "tenderness" of this oil in the presence of chemical reagents. The so-called qualitative tests for oils with sulphuric and nitric acid, etc., fail to give with maize oil reactions sufficiently characteristic to warrant their application in identifying or detecting this oil. Hence its bromine and iodine absorptions, its high Maumené figure, and the ease with which it saponifies in the cold, must be looked upon as the feature by which the oil may be best recognized.

Specimens of the potash and soda soaps were prepared, and both of these proved to be of good quality, being light in color, and readily and completely soluble in water. The soda soap is distinctly the harder of the two, but the potash soap is harder than the average "soft" soaps. Soap-making, therefore, is a use to which the oil might with advantage be put. The ease with which it

saponifies, also, might make it useful to mix with other oils to accelerate their saponification. As a lubricant it might in certain cases be applicable, its low acidity, and its small tendency to deposit solid matter or to "gum" being properties that recommend it for this purpose. The oil dissolves readily in acetone and more sparingly in alcohol or glacial acetic acid.

The viscosity was determined roughly by observing the time of flow of 5 cc. of the oil through a burette with a capillary point, and comparing the results with standard oils.

$t = 18°$ to $19°$ C.

| | Maize. | Olive. | Colza. | Mineral "901" |
|---|---|---|---|---|
| Specific gravity, | 0·924 | 0·918 | 0·915 | 0.910 |
| Time of flow, | 177·3″ | 244·5″ | 290·0″ | 243·5″ |
| Viscosity, | 61·1 | 84·3 | 100 | 83·9 |
| (Colza = 100) Colza (water = 1), | 25·7 | 35·4 | 42·0 | 35·2 |

Maize oil thus possesses a striking individuality. In general, it may be said that in properties it is somewhat akin to cotton-seed oil. At the same time there are differences between them which are very marked.

## THE USEFUL VARIETIES OF NUTMEGS.[1]

By Dr. Warburg.

The oldest works making mention of the nutmeg speak of several kinds which must have attracted the attention of the earliest Dutch travellers. In 1596, before the conquest of Banda, Linschoten mentioned two kinds of nutmeg—round and long—and in 1605, Clusius gave drawings of a fruit branch of *Nux myristicamus*, as well as the ordinary nutmeg.

In several of the older drawings of Piso and Valentini the leaves of the true nutmeg are incorrectly associated with the fruit of this second variety. The term Pala metsiri, often used by Piso, is probably also based upon a confusion of the true nutmeg with a variety. However, it is on the whole clear what is meant, and there is no

[1] Read at the meeting of the Berlin Pharmaceutical Society, June 2; reprinted from *Phar. Journal and Trans.*, July 2, 1892, p. 11.

doubt that all these remarks apply to the *Myristica fatua*, Houtt., a tree which certainly grows wild in Banda and Amboyna, perhaps, also in other of the Molucca Islands. Its fruit is scarcely at all aromatic when fresh, in the dry state not at all, while the mace smells disagreeable and not aromatic. The fruit is, therefore, only used medicinally in that island for dysentery, headache, or as an aphrodisiac, and they were brought from thence only as curiosities.

As the intercourse between the Moluccas and New Guinea increased, an entirely new kind of nutmeg came into the hands of Europeans, the *Myristica argentea*, Warb. That was probably first noticed in 1666. Since the middle of the 18th century it became an article of commerce in Eastern Asia. Towards the end of that century it was brought to Europe, and at the present time it is the most important article of export from New Guinea. Nevertheless this nutmeg remained undescribed and unknown, as well as the plant by which it is produced.

Warburg succeeded in obtaining information on this point through the assistance of a native who was persuaded to show him some of the trees in Dutch New Guinea. They were characterized by large leaves having a silvery appearance at the under side, and hence the name.

Next to *Myristica fragrans* the *M. argentea* is certainly the most important variety, and that which has the greatest future. The odor is not so delicate as that of the true nutmeg, but that may be due to the circumstance that it is not prepared and packed with as much care as the true kind. The export from the province of Onin is estimated by Beccari to have amounted to about 125 pounds at the middle of the 18th century, and it exceeded in importance that of all other produce. Since then the regular service of steamers has led to a great increase in this trade. Formerly, the nutmegs were sent in small parcels by ships to Banda, there treated in the same way as true nutmegs and sometimes mixed with them. Now they are all taken direct to Macassar where they are shelled and dusted with lime. The price of them in Macassar is about one-third that of the best quality of true nutmegs.

While formerly these nutmegs were used only in the Malay Archipelago, in the Philippine Islands, etc., by the natives, probably on account of their cheapness, as *Para papua* in the Malay country, as *Aniz moscada* in the Philippines, and came only occasionally to

Holland and England, they are now regularly imported by way of Amsterdam into England as long nutmegs, and they have been known in Germany since 1890 as horse nutmegs. Apart from the fact that the aroma is not so delicate the nutmegs are also very friable, but the broken fragments can be used for the production of essential oil. They are also very liable to be attacked by maggots even when they have been limed. The aroma is very permanent even when the nutmegs have been kept for a number of years. Samples dating from the previous century have still a strong smell when crushed.

Hitherto, the mace has not been brought into commerce. Samples of it brought to Europe have a dirty gray to brown-red color, but this is probably due to defective drying, since some of the nutmegs brought over by the author have a fine dark arillus that is very oily, and has a powerful odor. It is uncertain whether in drying the mace would acquire the yellowish red color of that from *M. fragrans*, but it is certainly capable of being made useful provided it can be properly prepared.

These nutmegs would come into actual competition with true nutmeg, only in the event of their being carefully cultivated and gathered, as the produce of *M. fragrans* is in Hainen, and it is not improbable that their lower price would be compensated by a larger yield

The nutmegs of *M. argentea* differ from true nutmegs in their narrow, long shape, and the relatively less marked arillus furrows. The arillus generally consists of four broad stripes, which are united above and below. The same with the hard shell is from 3½ to 4½ cm. long and from 2 to 2½ broad. It is broadest at the base and becomes gradually narrower towards the end, externally of a bright red-brown color when fresh, but as met with in commerce it is generally rubbed and of a yellow-brown color. The fruit is imbedded in a very thick pericarp, and when fresh it is from 4½ to 6½ cm. long and 4½ to 5½ cm. broad. The testa is nearly 1 mm. thick. The endosperm contains much starch, and the brown runcination streaks, which alone contain the aroma, are more scattered and coarser than in true nutmegs. The cotyledons are joined in a disc swelled at its edges to 5 mm. diameter.

Among other available kinds of nutmegs, the author mentioned *M. succedanea*, Reinw., discovered by Reinwardt in the island of

Tidore, one of the Moluccas, in 1821. The nutmegs can scarcely be distinguished from those of *M. fragrans*, and they are very aromatic. The leaves and flowers of this variety are, however, quite different from those of *M. fragrans*.

In New Guinea there is a great number of varieties of nutmeg plants, the produce of which possess some aroma, but though permanent it is generally too feeble to admit of these kinds being used to any extent as substitutes for true nutmegs.

As an adulteration of true mace the arillus of *M. malabarica*, Lam., known under the name of Bombay mace, has been used during the last two centuries. It is much larger and more cylindrical than the arillus of true nutmeg, and the several flaps are united at the apex, forming a conical structure. The anatomical structure is also different, as may be seen by the aid of a microscope. When moistened with hydrochloric acid the Bombay mace presents the marked peculiarity of assuming a greenish color.

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## "ON THE BEHAVIOR OF GALLIC AND TANNIC ACID IN THE ORGANISM."[1]

By Dr. C. F. Mörner.

It has been long known that when gallic acid is taken internally it can be found unchanged in the urine, and Wöhler and Frerich stated that when tannic acid is ingested it is excreted in the urine, as gallic and pyrogallic acid. Lewen afterwards showed that in the rabbit at least some tannic acid is met with in the urine after its ingestion as well as gallic acid, and six years ago Stockman proved that whilst in man some of the gallic acid taken passes unchanged, some of the tannic acid is excreted in the urine as gallic acid, and neither pyrogallic acid nor tannic can be found in the urine after tannic acid has been taken. In dogs and rabbits, however, the tannic acid is excreted, partly as gallic acid and partly unchanged, or at least in combination with alkalies. Mörner has made investigations which completely confirm Stockman's results, and has further estimated the amount of gallic acid present in the urine after definite doses of gallic and tannic acids. For this purpose he has employed a modification of a plan similar to that suggested by Wolkow and Baumann for the

[1] *Zeits. für Physiol. Chemie*, XIV, Heft 4 and 5; reprinted from *The Medical Chronicle*, July 1892.

estimation of homogentisinic acid in the urine, which depends on the reducing action of gallic acid on ammoniated silver solution.

He finds that the proportion of gallic acid excreted in the urine largely depends on the amount given in one dose. If a drachm to a drachm and a half be taken 30 per cent. passes in the urine; if 30 grains only 21 per cent. After 22 grains only 5 per cent. could be detected; after 7–15 grains only 2 per cent., whilst a dose of 3 grains was not followed by the appearance of any gallic acid in the urine. As he examined the fæces in vain for any trace of gallic acid it follows that a certain portion is burnt up in the system in its passage through the body. He finds that after tannic acid has been taken only very little gallic acid passes into the urine. The administration of 30 grains to 60 grains of tannic acid is indeed followed by the appearance of a trace of gallic acid in the urine, but so small is the quantity that he could not estimate it. Only when two drachms of tannic acid had been administered was the quantity of gallic acid sufficient to allow of its quantitative estimation, but only one per cent. of tannic acid taken was found in the urine as gallic acid; since tannic acid could not be found in the fæces he concludes that the greater part of the tannic acid which is absorbed is burnt up in the body.

In explanation of the very small quantity of tannic acid which appears in the urine, changed into gallic acid, he suggests that the former forms with albumen, combinations difficult of solution; these pass into the intestine, and there slowly decompose. The tannic acid is, indeed, converted into gallic acid, but only gradually, and the small quantities of the gallic acid thus produced are burnt up and therefore, never appear in the urine. On the other hand, when gallic acid is taken, owing to its great solubility it is absorbed, and entering the blood at once, a small quantity only is burnt up and the larger quantity excreted.

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## MOLYBDIC ACID AS A COLOR REAGENT FOR CERTAIN AROMATIC OXY-COMPOUNDS.

By J. Stahl.

Hager some time ago indicated a reaction for the tannic acid of galls and other tannic acids, according to which these substances give fine reddish-yellow colors with ammonium molybdate. I have found that the same reactions occur for certain compounds

approximating on tannin, pyrogallol, pyrogallo-carbonic acid, and gallic acid. As all four substances named contain oxy-groups in an ortho-position to each other, the reagent was tried also for other aromatic compounds in which the same case occurs. The result was that ammonium molybdate is a specific color reagent for all aromatic compounds which contain two or more oxy-groups standing in the ortho position to each other.

Ammonium molybdate produces intense phenomena of coloration only with such bodies, which, according to the substance employed, may be yellow, reddish-brown, or blackish-brown. On the contrary, in all organic compounds in which the above-mentioned condition does not occur no coloration is recognized. Thus, with pyro-catechin on the addition of the molybdate there is shown an intense reddish-brown color, which does not appear with hydroquinone and resorcine—a behavior which shows phloroglucine in contrast to pyrogallol. The following aromatic compounds display similar colorations: Protocatechuic acid, caffeic and hydrocaffeic acid, phenanthrenehydroquinone and retenehydroquinone; in alizarin, purpurin, anthragallol, and rufigallic acid, the aqueous solutions of which are yellow or red, the tone is much intensified by an addition of the molybdate. The entrance of other groups into the nucleus does not generally interfere; thus the reaction appears in the bromo and nitro derivatives of pyrogallol and gallic acid, but not in the phenol ethers, where one or more hydrogen atoms of the oxy-group are replaced by alkyles, as guaiacol and vanilline. Very slight yellow colorations which may occur with the two last-mentioned bodies are probably due to slight impurities. Among compounds of a less fully known constitution the reaction is produced by quercitrin and its scission product quercetin, apomorphin, podophyllin, koussein and aloin.

The aqueous solution of free molybdic acid behaves exactly like ammonium molybdate. The latter, in presence of organic substances, passes to a lower stage of oxidation, with the production of a blue, yellow, or a reddish-brown.

The origin of the color phenomena in question depends on the oxidizing action of molybdic acid and its ammonium salt. As those aromatic compounds which contain oxy-groups in the ortho-position to each other are more unstable and especially more readily oxidizable than their isomers, there occurs here a more

energetic reduction of the molybdic compounds. The stage of the blue oxide is overleaped, and there appear at once the lower brownish stages of oxidation. If heat is applied and the reaction is prolonged, brownish-black masses separate out which are found to be a variable mixture of lower molybdic acids, whilst a portion of the organic substance is oxidized to carbonic acid and water. But it is not impossible that there may appear simultaneously yellow or brown oxidation products of the oxy-compounds.

The sensitiveness of these reactions is not very great, but in case of pyrocatechin, pyrogallol, gallic acid, and tannin, it admits of the recognition of 0·1 mgrm. substance in one cc. of liquid. The action of sodium tungstate is similar to that of ammonium molybdate.—*Chem. News*, June 24, 1892, p. 302.

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## THE FLAME OF BURNING NITROGEN.

By W. Crookes, F.R.S.

Nitrogen is a combustible gas; that is to say, a mixture of nitrogen and oxygen (atmospheric air), will under certain conditions burn with a flame, and production of nitrous and nitric acids. The reason why, when once nitrogen is set on fire, the flame does not spread throughout the whole atmosphere and deluge the world in a sea of nitric acid, is that the igniting point of nitrogen is higher than the temperature produced by its combustion, and therefore the flame is not hot enough to set fire to the adjacent gas.

In the experiments shown at the Soirée of the Royal Society, on June 15, an electric current of 65 volts and 15 ampéres, alternating 130 times a second, was passed through the primary of a large induction coil, when an arching flame, consisting chiefly of burning nitrogen, issued from each of the secondary poles, meeting at the centre. When once started the poles can be drawn asunder till the flame bridges across 212 mm. When the terminals are more than 46 mm. apart, the flame will not strike across. When alight the flame is easily blown out by the breath, and it can then be re-lighted by a taper.

In the spectroscope the flame of nitrogen shows no lines, the spectrum being faint and continuous. The temperature is a little higher than that of a good blow-pipe flame, easily melting fine platinum wire. The hot gases rising from a flame have a strong

odor of nitrous acid, and when it is produced in a closed globe, the interior rapidly fills with red gases.

The flame produced by exciting an induction coil by means of an alternating current was first observed by Mr. Spottiswoode, F.R.S., who described it before the Royal Society in 1880. It has lately been exhibited on a magnificent scale at the Crystal Palace, by Messrs. Siemens Bros., and by Messrs. Swinburne & Co. It is not known, however, that any chemical explanation of the flame has before now been published.—*Chem. News*, June 24, 1892, p. 301.

## THE SEPARATION OF SALICYLIC ACID FROM BENZOIC ACID.

By Miss J. Schaap.

There does not at present exist a good quantitative process for the separation of these acids, and hoping to effect this I first tried the precipitation with alum. I found, however, that the aluminum benzoate is not quite insoluble in water, so the method is therefore valueless for quantitative purposes. Better results were obtained by the following method:

A few mixtures, each consisting of 0·25 grm. of salicylic and 0·25 grm. of benzoic acid, were dissolved in a sufficiency of hot water and the liquids were then allowed to cool. The salicylic acid was now precipitated by excess of bromine water, but the question had to be decided whether it was precipitated as the mono-, di-, or tri-bromo compound. I, therefore, took 0·25 grm. of the bromo-salicylic acid and estimated the bromine by Fresenius' lime method. The nitric acid used was free from chlorine, but the lime was not, so a check had to be made. Allowing for the small quantity of chlorine in the lime, the results in four experiments were, respectively: 0·305, 0·315, 0·319 and 0·314 grm. of AgBr. Theory requires 0·215 grm. for the mono-compound, 0·316 grm. for the di-compound, and 0·374 for the tri-compound; so it follows that the precipitate consisted of the di-compound.

Another question now arose whether the precipitate was sufficiently insoluble in water, and also whether the precipitation was complete. To solve this question 0·25 grm. of salicylic acid was dissolved in water and precipitated with bromine water. Having found that the bromo-compound is very readily soluble in chloroform, I concentrated the filtrate at a temperature of 30° and agitated

the liquid with chloroform, which on evaporation did not yield the smallest residue, showing that all the salicylic acid had been precipitated. The precipitated dibromo-salicylic acids were dried in a desiccator and afterwards weighed. The weights were, respectively: 0·56, 0·556, 0·551 and 0·553 grm., theory requiring 0·536 grm.

In order to directly estimate the benzoic acid, the filtrates were rendered faintly alkaline with sodium carbonate, and evaporated to a small bulk on the water-bath to expel the excess of bromine. The residues were then put into separatory funnels, acidified with hydrochloric acid, and shaken out with chloroform. This was filtered through a dry filter and allowed to spontaneously evaporate in weighed glass dishes. The results were, respectively: 0·272, 0·261, 0·237 and 0·232 grm. of benzoic acid, instead of 0·250 grm.—*Ned. Tydschr. v. Pharmacie, etc.*, July, 1892; Chemical News, vol. 66, p. 43.

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## AMERICAN PHARMACEUTICAL ASSOCIATION.

Most of the members and delegates who attended the fortieth annual meeting of the American Pharmaceutical Association, followed the advice of the local secretary and the Committee on Arrangements, that Boston be considered the gathering point for those whose route of travel would permit.

On Saturday, July 9, the members began to arrive there, nearly all stopping at the Hotel Vendome, Commonwealth Avenue and Dartmouth Street, where elegant quarters had been provided. A Committee of Entertainment and a number of special committees, representing the pharmacists and druggists of Boston and Massachusetts, took charge of the visiting members and their ladies, and were unremitting in their attentions. For Monday, July 11, excursions had been planned to Bunker Hill Monument and Charlestown, to Cambridge and Harvard University, to Concord, Lexington, Plymouth and Salem, each one being conducted by a special committee. Tuesday was set apart for a harbor excursion on the steamer *City of Jacksonville*, starting from Battery Wharf and landing at Bass Point, where a sumptuous fish dinner was served. Returning to the city shortly after noon, the remaining portion of the day was devoted to a carriage drive, starting from the Vendome at about 3 o'clock. Many places of interest in the city and suburbs were visited, and in the evening a delightful reception was tendered to the visitors at the Vendome, with music and refreshments, the company separating towards midnight. On the following morning, Wednesday, the journey to the place of meeting was undertaken in a special train, which landed the excursionists at Alton Bay on Lake Winnipisaukee, where a steamer was in waiting, with a committee of the New Hampshire pharmacists, who vied with their Massachusetts brethren in entertaining the visitors while on their way to the Profile House. Music had been provided, as well as an excellent luncheon and other enjoyments, and before the company disembarked at Weirs, President Finlay gave expression

to the high appreciation of the constant courtesies shown, closing with a proposition for three cheers for the generous hosts, which was heartily endorsed, and ardently carried out. The last portion of the trip was made by rail through the Pemigewasset Valley to Bethlehem Junction, and thence to the Profile House, arriving there about supper time. The arrangements had been made with such foresight that, on arrival at the hotel, every one of the party found the baggage, which had been sent by a different route, already in the room assigned for his or her occupancy during the next five or six days.

After supper a meeting, lasting until after midnight, was held by the Council to arrange the business for the Association.

*First Session, Thursday Morning, July 14.*—Shortly after ten o'clock the meeting of the Association was called to order in the large parlor of the Profile House, when President Finlay introduced Mr. A. P. Preston of Portsmouth, N. H., who, in behalf of the pharmacists of New Hampshire, spoke words of welcome and of best wishes for a successful meeting, and after referring to the grand scenery and the varied attractions in the State, briefly outlined the program of entertainment following the meeting, and expressed the appreciation to the ladies for the part they had taken in New Orleans in suggesting the White Mountains as the place for meeting, closing by saying: "We thank you sincerely, ladies; you have shown excellent judgment and your husbands excellent taste in following your suggestion." Vice-president Tobert was called upon to reply, which he did in a happy manner, referring to the generous hospitality, pledging return as occasion may offer, alluding to the community of interests between pharmacists everywhere and the American Pharmaceutical Association, and closing with a reference to the influence of New England upon the growth of the republic.

President Finlay, in his presidential address, called attention to the fact that this very day was the one hundredth anniversary of the first president of this Association, Daniel B. Smith, who was born in Philadelphia, July 14, 1792, and in October, 1852, was elected president of the National Pharmaceutical Convention, and of the American Pharmaceutical Association then organized. Mr. Finlay then passed in review the action of the representatives of the Association during the past year, referring to the address of congratulation sent to the Pharmaceutical Society of Great Britain; to the conference with the National Wholesale Druggists' Association relating to the fixation of prices for proprietary articles; to the section of Materica Medica and Pharmacy of the American Medical Association, and to the invitation extended for holding the Seventh International Pharmaceutical Congress in Chicago. The progress in the revision of the U. S. Pharmacopœia was alluded to, and reference was made to the continually increasing number of synthetical remedies, some of which proved to be material quite unremunerative on the necessary investment. In closing, allusion was made to members who have faithfully served the Association, aiding in its counsels and enriching its literature, and who have given the results of their patient labor and inventive genius, and it was suggested by the President, that to them some token of appreciation, some tribute of esteem was due, which might be in the form of an emblem bearing an appropriate motto, or a scroll suitably inscribed.

The address was referred to a Committee of three, which subsequently reported on the suggestions, and approving of the last one, recommended its

reference to a Committee of five for formulating a plan embodying the president's idea.

The list of delegates, which was reported by the Council, showed that delegations had been appointed from

*Colleges of Pharmacy:* Chicago, Cincinnati, Denver, Illinois, Louisville, Maryland, Massachusetts, National (Washington), New York, Philadelphia and St. Louis;

*State Pharmaceutical Associations:* Alabama, Arkansas, Colorado, Connecticut, Delaware, Florida, Georgia, Illinois, Indiana, Iowa, Kansas, Kentucky, Louisiana, Maine, Maryland, Massachusetts, Michigan, Missouri, New Hampshire, New Jersey, New York, North Carolina, North Dakota, Ohio, Oregon, Pennsylvania, Rhode Island, South Carolina, South Dakota, Tennessee, Texas, Virginia, Washington, Wisconsin, Nova Scotia and Quebec;

*County and City Associations:* Cleveland, O.; Houston, Tex.; Kings Co., N. Y.; German Apothecaries, New York;

*Alumni Associations of Colleges of Pharmacy:* Cincinnati, Maryland, Massachusetts, New York, Philadelphia and St. Louis.

The Secretary of the Council presented the names of 289 candidates for membership. Intimation being made that objections might be made to one of the candidates, voting on the admission was for the present postponed.

Reports of Committees were called for and laid upon the table for future action.

A telegram was received from Dr. Henry O. Marcy, President of the American Medical Association, conveying the hearty sympathy of that Association, and regretting that he had been unexpectedly detained.

The appointment of the Nominating Committee was effected in the usual manner, one or two representatives being named from each State or Province; the representatives of six other States arrived too late to be represented on this Committee. Messrs. Diehl, Ingalls, Fennel, Keppler and Ramsperger were appointed from the association at large.

The Secretary of the Council read the minutes of that body since the New Orleans meeting, which were approved. Besides the business transacted in the interim between the two meetings, and the arrangement of the business for the present meeting, various reports of officers and committees had been examined and passed upon.

The Committee on Membership reported the decease of 29 members and 2 honorary members during the preceding year.

The Committee on Publication recommended that the plan, inaugurated last year, of furnishing each member with a printed copy of the minutes, in pamphlet form, and in advance of the bound copy of the Proceedings, be continued in the future. Quite a number of partial and complete sets of the Proceedings have been sold during the past year, realizing $271.15, and these sales have reduced several of the older issues to such an extent that they are likely to be soon completely out of print.

There is still a steady demand for the National Formulary; during the eleven months ending May 30, 1892, the cash receipts from this source were $799.40, and the expenses $292, making the total cash profit to the Association, since 1888, amount to $2,660.71, aside from the 1,421 copies which have been furnished gratuitously to the members and others.

Including cash balance on hand at the beginning of the last fiscal year, amounting to $6,670.33, the Treasurer reported the total receipts for the eleven months to have been $12,962.75, and the total disbursements $9,128.42, the latter sum including $3,021.62 for permanent investment, leaving a balance on hand, June 1, of $3,834.33, which was decreased July 1 to $3,703.25.

The invested funds in the custody of the Chairman of the Council are as follows:

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| Ebert fund, | Cash, | $62 25 | and bonds | $600 | valued at | $699 00 |
| Centennial fund, | " | 188 99 | " | 1,100 | " | 1,282 00 |
| Life membership fund, | " | 772 47 | " | 8,100 | " | 9,436 50 |
| General fund (see Treasurer's report), | | | " | 3,000 | " | 3,000 00 |

Prof. Whelpley gave notice of an amendment to Chap. iv, Article iv, of the by-laws, increasing the Treasurer's salary from $600 to $750, which was adopted at a subsequent session.

After the appointment of a Committee on the time and place of the next annual meeting, consisting of Messrs. Sheppard, Remington, Main, Sloan and Ebert, the Association adjourned until 4 o'clock, P. M.

*Second Session, Thursday afternoon.*—After the reading and approval of the minutes, the Nominating Committee submitted the following nominations for officers for the next year: President, Jos. P. Remington, Philadelphia; Vice-presidents, A. P. Preston, Portsmouth, N. H.; S. P. Watson, Atlanta, Ga., and W. H. Averill, Frankfort, Ky.; Treasurer, S. A. D. Sheppard, Boston; Permanent Secretary, J. M. Maisch, Philadelphia; Reporter on the Progress of Pharmacy, Henry Kraemer, New York; members of Council, H. M. Whitney, Lawrence, Mass., Gust. Ramsperger, New York, and Chas. E. Dohme, Baltimore. In regard to the office of reporter it was stated that Dr. Rice had positively declined a re-election, because he could not give to the work the personal attention which it ought to receive; also, that Mr. Kraemer has made, under Dr. Rice's supervision, a portion of the abstracts and compilations for the report of the current year. All the nominees were duly elected.

The Committee on International Pharmaceutical Congress presented a report through the chairman, Prof. Oldberg, in relation to measures to be taken for the Seventh International Congress, to be held in Chicago next year, all of which were referred to the Council with power.

Prof. Diehl made a report for the Committee on National Formulary, discussing the popularization of the Formulary and its revision under the headings of corrections, additions and eliminations. The report was accompanied by a lengthy epitome of criticisms on the present National Formulary, arranged in three parts, giving abstracts from journals, a synopsis of communications from members of the Committee, and the results of examination of typical specimens of National Formulary preparations, after having been kept on hand for nearly four years. The report shows that the formulas of the National Formulary have proved to be very satisfactory, that very few alterations have been proposed, that very few additions have thus far been suggested, and that the preparation of a final report cannot be undertaken until after the publication of the new pharmacopœia.

The report of the Committee on time and place of meeting, recommended Chicago, the time to be designated by the Council, which was adopted.

Amendments lying over from last year were then considered. The amend-

ment to Art. iv of the Constitution, changing the word shall to may, created much discussion, but was finally tabled, when it was shown that the word shall was not mandatory, but merely directory, and that the interest from life membership was to go into the common account, merely in lieu of the annual contributions of contributing members.

Chapter ix, Article viii, of the by-laws, was amended so as to give two sessions to the Section on pharmaceutical legislation and education. The proposed amendment to Chapter viii, Art. v, to receive delegations from State pharmaceutical associations only, was laid on the table.

A communication from Mr. Abernethy, relative to the publication of an official dose book, was read and referred to the Section on scientific papers.

*The Section on Commercial Interests* held its sessions on Friday, July 15, the chairman, W. H. Torbert, presiding; A. Bassett, Secretary. The Committee appointed at New Orleans reported that they had been present at the meeting of the National Wholesale Druggists' Association at Louisville in October, 1891, and conferred with that Association and the Association of Manufacturers and Proprietors with the result that the plan for the prevention of cutting of prices elaborated at New Orleans was adopted by the two Associations named, with slight modifications, and that a committee had been appointed, consisting of members of the three Associations, with the view of working out the details and taking steps to put the plan in operation. The chairman in his address stated that while the plan had been very generally approved, even by lawyers in many States, some lawyers considered it to be in conflict with the anti-trust laws, and that this opinion had been a stumbling block in the way of its successful and general adoption.

Letters referring to the same subject were read from a Committee of the Michigan State Pharmaceutical Association, from the president of the Interstate Retail Druggists' League at St. Louis, and from the Illinois Pharmaceutical Association.

A communication from the Colorado State Pharmaceutical Association urged that efforts be made for securing the repeal of the annual tax of $25 imposed upon druggists for the purpose of retailing liquors for medicinal use and distilled spirits for mechanical purposes.

A communication from the Arkansas Association of Pharmacists suggested a memorial to the Congress, urging the repeal of all copyright laws which protect the names or the process of manufacture of any and all chemicals and remedies used for the relief of human suffering which are put up and recommended to the use of physicians and others, no matter under what name or form, except those agents known as patent or proprietary remedies.

The discussion took a wide range, and it was finally concluded that a Committee of three consider the subject and report at the final session to the Association, which was done by recommending that the proprietors put the A. P. A. plan in force promptly, and omit the delay in the submission of it to the retailers for approval. Messrs. Torbert, Bassett, Hechler, Rano and Canning were appointed a special committee of the Section for the prevention of cutting, and the same committee was also recommended to the Association for appointment as delegates to the National Wholesale Druggists' Association.

Among the resolutions passed by the Section were the following:

That all manufacturers of medicinal preparations be requested to devise simple methods of marking all packages for retail use so as to facilitate determining the source of supply of such goods.

That the Association urge thorough local organization in every centre for the purpose of supporting the A. P. A. plan; also,

That it be requested that the proceedings of the commercial Section be specially printed as soon as possible in pamphlet form, and be sent to every member of the Association.

The Section re-elected its officers for the ensuing year: W. H. Torbert, chairman, and A. Bassett, secretary, and added Messrs. C. O. Rano and G. L. Hechler as members of the committee.

*The Section on Scientific Papers* held three sessions on Saturday, July 23, Prof. Hallberg in the chair; in the absence of Secretary Snow, Professor Fennel acted in that capacity. The following papers were read:

*Chemical Symbols.*—W. W. Kerr, Batesville, Ark., suggested that in addition to the names of chemicals also the chemical formulas be placed upon the labels of the containers.

*Examination of the Amyl Nitrite of Pharmacy.*—Prof. Curtman read an elaborate paper on this subject, exhibited the apparatus and showed the practical working of the process, which is an adaptation of that devised by Allen for spirit of nitrous ether, while the instrument is a modification of Lunge's nitrometer. The method is based upon the conversion of amyl nitrite, by the addition of potassium iodide and sulphuric acid, into amyl alcohol, potassium bisulphate, free iodine and nitric oxide (NO or $N_2O_2$), one molecule (116·78 gm.) of amyl nitrite yielding one molecule (29·97 gm. = 22321·2052 cc.) of nitric oxide gas; or 0·5231797 gm. of the former yield 100 cc. of NO. The analysis is made by filling the nitrometer, including the bore of the stopcock at the top, with a saturated aqueous solution of sodium chloride; 0·523 gm. of the amyl nitrite diluted with about 5 cc. of alcohol, is now added through the stopcock with the precaution of not admitting any air, and of washing down the last portions of the nitrite with a few cc. of the salt solution. The reagents are used in the strength of normal volumetric solutions, 10 cc. of KI solution being introduced first, followed by 10 cc. of normal $H_2SO_4$. A strong effervescence takes place, but the reagents being specifically lighter than the salt solution, some agitation is necessary to complete the reaction which is usually accomplished in about five minutes. The nitrometer tube being connected by means of a rubber tube, with an open equilibrium tube, the latter is now lowered until the liquid in it is about 3·3 cc. lower than that in the measuring tube, when the number of cc. of NO gas is read off and noted. The correction for temperature must be made by dividing the number of cc. by the volume in cc., to which 1 cc. of NO, measured at 0° C., will expand at the temperature prevailing at the time of making the experiment (the increase for each degree C. = 0·03663 cc.); the correction for pressure is made by multiplying with the present pressure in mm., and dividing by 760 mm., the normal pressure—or by multiplying with the present pressure in inches and dividing by 30. The number of cc., thus corrected, gives the percentage of amyl nitrite in the specimen.

Examining fifteen samples by this method, four were found to contain between 27·14 and 39·60 per cent., one 59·33 per cent., three over 60 per cent.,

three over 70 per cent., three over 80 per cent., and one 93·71 per cent. of amyl nitrite. The preparation must be preserved with the utmost care, and particularly protected from light. A specimen assaying 65·35 per cent., after having been kept on the prescription desk for three months contained only 39·60 per cent. of amyl nitrite; and another sample assaying 84·235 per cent., after one week's standing in a white glass vial was reduced to 71·32 per cent. amyl nitrite. The nature of all the impurities present was not determined; one sample contained much amyl valerianate and some butyl nitrite. Attention is directed by the author to the fact that the addition of methyl alcohol to amyl nitrite converts the mixture into amyl alcohol and gaseous methyl nitrite; and that the addition of ethyl alcohol gradually results in the production of ethyl nitrite and amyl alcohol.

*Economic Percolation* was the title of a paper presented by Harry Vin Arny, in which the results of numerous observations were given, showing the loss of alcohol by evaporation in conducting the process of percolation. In estimating this loss the amount of percolate was taken into consideration, and the marc remaining in the percolator was subjected to distillation in order to recover the alcohol as far as practicable. To guard against this loss or reduce it to a minimum, an air-tight percolator was constructed, resting upon a receiver, terminating below with a tapering bottom, provided with a straight faucet; the two parts are fastened together by means of a screw joint, and further connected by means of a tube extending from the top of the percolator to the top of the receiver. In order to facilitate the withdrawal of the percolate from the faucet, the top of the receiver is provided with an additional orifice through which air is admitted, but which is closed with a screw cap while percolation is going on.

*The separation of strychnine and brucine* was discussed in a paper by H. W. Snow. Various processes for estimating the strychnine in mixtures of the two alkaloids were tried without satisfactory results. On treatment with nitric acid, sp. grav. 1·056, it was found that a portion of the strychnine was destroyed. The author regards the subject as a most discouraging one, and though he has performed many experiments, has as yet nothing but negative results to offer. It should, however, be stated that the process suggested by Gerock (*Amer. Jour. Phar.*, 1889, p. 180) has not been tried by Mr. Snow.

*Aromatic spirit of ammonia.*—A. Conrath observed that using ammonium carbonate picked out of a cask opened for the purpose, a preparation could be made by the pharmacopœial process, without containing a permanent precipitate. Using the carbonate as ordinarily met with, a precipitate usually occurred, even after increasing the quantity of ammonia water, if the alcohol had been added at once, but on leaving the alkalies together for several hours before adding the alcohol, no precipitate was formed. The author suggests the use of bicarbonate of ammonium as leading to greater uniformity in the strength of the preparation.

*American Potash.*—Prof. Lloyd has found commercial potash of very uneven quality, and the commercial terms of first, second and third sorts to have no definite significance, since the poorest potash is sometimes sold as first sorts; the adulteration consists chiefly of table salt and lime. In the casks examined the carbonate and hydrate combined assayed sometimes as low as 16·5 per cent. KOH, while casks were met with assaying sometimes as high as 91 per cent.

KOH. The author believes that if a standard of at least 70 per cent. KOH were authoritatively established, low grade potash would then be prevented from entering the general market, and the product would be improved.

*The juice of taraxacum.*—Prof. Sayre collected the root May 10; drying it at 45° it lost 79 42 per cent. of moisture, and this on drying at 100°, lost further 10·68 per cent. The fresh root yielded by pressure 57 per cent. of juice, of spec. grav. 1·007, and containing total solids 1·472, sugar 0·036, and ash 0·0045 per cent.

*Oil of Wintergreen* is proposed by B. H. Ewing, to be assayed volumetrically by saponifying 5 gm. of the oil with 40 cc. volumetric solution of soda at 60° C.; after the precipitate formed at first has totally disappeared, cool the flask, then boil for five minutes, again cool, add phenolphthaleine, and titrate with normal hydrochloric acid until the red color disappears; subtract the volume of acid required in cubic centimetres from 40, multiply the remainder by ·138 ($\frac{1}{1000}$ molecular weight of salicylic acid) and the resulting product by 20 to get the percentage of salicylic acid. Methyl salicylate is obtained by using in the calculation the figure ·152 in the place of ·138. The results agree closely with those obtained by determining the salicylic acid gravimetrically. Genuine oil of gaultheria assayed 90·15 salicylic acid and 99·30 methyl salicylate. Genuine oil of sweet birch yielded 90·20 acid and 99·40 of the methyl compound. In an adulterated specimen only 68 per cent. of methyl salicylate was indicated by this method.

*Solution of bimeconate of morphine.*—A commercial sample examined by Alice L. Braunwarth yielded 0·908 per cent. solid residue containing 0·80 morphine, 0·085 meconic acid and 0·091 HCl. The solvent was diluted alcohol of 24 per cent. by weight.

*The botanical names of the U. S. Pharmacopœia* have been examined by Professor H. H. Rusby, in accordance with the rules adopted by the International Botanical Congress, held at Paris in 1867, according to which no name has a right to supersede the first name properly given to a plant since the binomial nomenclature was adopted by Linnæus. In an elaborate paper presented by the author, the names and synonyms are given and the authorities cited with the dates of first publication. If the rule is to be strictly followed, a number of botanical names will have to be changed.

*The chemistry of the elements entering into Syrup of phosphates of iron, quinine and strychnine, U. S. P.*, by Professor Fennel, is an inquiry into the causes of the changes produced in this pharmacopœial product, and he arrives at the conclusion that with the exercise of the greatest skill and judgment the precipitation of basic ferric phosphate cannot be prevented and that the formation of this basic salt is the result of natural influences; further, that the precipitation may be accelerated by the careless preparation of any one of the compounds which enter into the final product, and that a lack of skill and judgment and the necessary precautions essential to the production of trustworthy compounds, apparently indicate a defective formula; yet such is not the case, for the formula is as perfect as man can make it.

*Calcium hypophosphite* was examined by Prof. Sayre by Moerk's method (see Amer. Journ. Phar., 1889, pp. 326 and 391), with potassium permanganate, the five samples assaying 96·92, 98·23, 99·16, 99·32 and 99·71 per cent., the balance being sulphate and carbonate.

*Syrup of calcium lactophosphate* was the subject of two papers by H. W. Aufmwasser, one giving the results of an analysis; the other containing a formula for its preparation, as follows: Dissolve calcium carbonate 21·3 p. in a mixture of phosphoric acid (50 per cent.) 109·4 p. and lactic acid 33 parts, previously diluted with orange flower water 80 p. and water 150 p., filter and wash with water to obtain 400 parts of filtrate, in which dissolve sugar 600 parts.

*The cultivation of coffee in Jamaica.*—This paper is reprinted on p. 396 of the present number.

*Reaction between borax, sodium bicarbonate and glycerin.*—Attention is again directed by Professor Lloyd to the decomposition which takes place between the two salts when in solution with glycerin, whereby one-half of the carbonic acid is liberated as gas. This reaction was explained in pharmaceutical and chemical journals in the years 1877 and 1878.

*Acid sublimate dressing.*—A. Levy suggests the preparation of compressed tablets containing mercuric chloride 3·5 and tartaric acid 17·5; these dissolve readily in warm water, and such solutions are well adapted for use as dressings. Solutions of corrosive sublimate are rendered inefficient as antiseptics by contact with albuminous and other organic matters; to counteract this effect, tartaric acid has been recommended (see *Am. Jour. Phar.*, 1888, 146, 404, and 1890, 554); also citric acid (*ibid.*, 1887, 355) and sodium chloride (*ibid.*, 1887, 396, and 1888, 407).

*The practical use of the microscope in pharmacy* was discussed in a paper by Dr. A. R. L. Dohme, of which an abstract cannot be made; the paper is illustrated by drawings of the transverse sections of pareira brava root and coca leaf.

Papers were also read on phosphate of iron and phosphoric acid, by L. F. Stevens; on glycerin in syrups, by L. F. Stevens; on the alkaloids accompanying berberine, by W. W. Birkiner; on the relationship of these white alkaloids, by R. D. Young; on the action of hot sulphuric acid upon bees-wax, paraffin and ceresin, and one entitled laboratory notes, by F. A. Thompson. Several of the papers presented had been prepared by non-members, and were accepted by formal vote and referred. Subsequently two papers were handed in by Prof. Kremers, entitled notes on queries and the menthol group.

The Committee on Prize Essays reported that the Ebert prize for 1891 be awarded to Prof. Lloyd for his paper, entitled "a scheme to establish a comparative standard for alkaloidal galenicals." A special committee for the examination of papers, marked "for competition" and read at the present meeting, reported in favor of awarding the first prize offered in 1887 ($75) to Prof. Fennel, and the second prize ($50) to H. V. Arny. These reports were adopted by the Section.

The officers of the Section for the ensuing year are: C. T. P. Fennel, Cincinnati, chairman; F. G. Ryan, Philadelphia, secretary, and Chas. Caspari, Jr., Baltimore, third member of the Committee.

*The Section on Pharmaceutical Legislation and Education* held its sessions on Monday, July 18, Prof. A. B. Stevens in the chair. In his annual address he referred, among other subjects, to a decision of the Supreme Court of Michigan affirming the constitutionality of that clause of the Michigan pharmacy law which prohibits physicians from conducting a pharmacy store without proving their qualification for it by passing the required examination before the pharmacy board.

Reference was also made to a bill which had been presented to the present Congress, requiring that the positions of apothecaries in the Navy be declared vacant, unless the present incumbent held a diploma from a College of Pharmacy or a certificate of examination from a State or County Board of Pharmacy, and that new appointments be hereafter made only from those who have graduated from a recognized College of Pharmacy. The Chief of the Bureau of Medicine and Surgery opposed this legislation, because it would summarily dismiss a large number of apothecaries who are entirely competent and who are doing faithful service, and because the present laws and regulations were entirely adequate; this view was concurred in by the Secretary of the Navy.

Secretary Hogan presented a lengthy report on the instruction given in the forty-eight colleges, schools, and college or university departments, existing in the United States and Canada; several of these have, apparently, only a nominal existence.

Professor Simon, in answer to a query, read a paper in advocacy of *extending the college course* in pharmacy to three years, but in opposition to the suggestion that the third year's course be devoted to physiology and therapeutics; there is ample room, and also necessity, for increasing the work in practical chemistry and other branches directly pertaining to pharmacy without encroaching upon the field properly belonging to the physician.

A paper by Prof. Sayre on *the teaching of pharmacy to medical students* suggests that this should necessarily be limited to the physical properties of preparations, and the general processes for making them, to their relative strength, to the chemical constitution of drugs and to their behavior in combinations. Regarding the therapeutical knowledge desirable for the pharmacist, the author thinks that the latter may advantageously know what remedy is applicable in certain classes of disease, but the physician must know definitely by practice the details of treatment in the use of the remedy.

A paper *on the Hospital steward in the U. S. Marine Hospital Service*, by L. A. Duckert, deals with the various duties of this officer, which comprise those of a competent pharmacist, and a good book-keeper, and require some executive ability and sufficient knowledge of mechanics to superintend repairs. The course to be pursued for admission to this branch of service is likewise indicated in the paper.

*Notes on Pharmaceutical Education*, read by Prof. E. Kremers, deal mainly with the study of materia medica. This term, in its broadest sense, embraces now several distinct branches of science like pharmacognosy, pharmaco-chemistry, pharmaco-dynamics, therapeutics and pharmaco-morphics (or pharmacopoetics). The author then outlined several methods for teaching pharmaceutical materia medica, and arrives at the conclusion that the salvation of the pharmacist does not lie in making all microscopists or all analytical chemists, but that it will be achieved by raising all to the highest educational standard prevalent in the country, and by allowing the individual to find for himself the special field for which he is best fitted.

A proposition made by Mr. Alpers, of Bayonne, N. J., that all applicants for examination by the Pharmacy Boards should have previously graduated from some reputable college of pharmacy, created a great deal of discussion, mostly in opposition. Since Mr. Alpers is neither a teacher, in nor a member of, a college of pharmacy, it seems evident that his proposition was made in

the interest of pharmaceutical education; it was withdrawn and no action taken.

A resolution offered by Prof. Fennel, that all reputable colleges of pharmacy be recommended to adopt a three years' course as soon as practicable, was adopted.

Prof. Robert G. Eccles, of Brooklyn, was elected chairman for the ensuing year, and L. C. Hogan, Chicago, was re-elected secretary.

*The final session* of the Association was held on Monday evening, when the names of additional candidates for membership were reported, making a total of 400 received at this meeting. The Nominating Committee proposed Prof. Hallberg for local secretary for the next meeting, but he declining, nominated Mr. Henry Biroth, who was duly elected.

Professor Remington read the report of the delegates to the Section of Materia Medica and Pharmacy of the American Medical Association, in which it was recommended that the president be authorized to tender an invitation to the American Medical Association to send a visiting delegation to the next meeting at Chicago. The recommendation was adopted.

Reports were also read from the Committee on the adoption of the metric system and from the Committee on Transportation.

Various amendments to the by-laws were then acted on and adopted, as follows: Chap. viii, Art. ii, a list of the names of the proposed candidates is to be posted in the meeting hall at the beginning of a session. Chap. ix, Art. iv, now permits also the proposition of amendments preceding the sessions of the different Sections. In Chap. vii, the word appointed, occurring in Articles II and III, was changed to elected. A communication received from Mr. A. Snyder, in reference to membership, was referred to the Committee on Membership.

A committee from the Section on Commercial Interests reported a recommendation "that the proprietors put the A. P. A. plan in force promptly and omit the delay in the submission of it to the retailers for approval." After some discussion, this was adopted.

Mr. Bassett reported from the same Section a request to appropriate sufficient money for defraying the expenses of the Committee visiting the National Wholesale Druggists' Association at its next meeting, and moved that the amount of such appropriation be subject to the approval of the Council. Carried.

On motion of Mr. Martin a sufficient sum was appropriated to pay for the prizes awarded by the Section on Scientific Papers.

A motion of Mr. Sheppard was adopted, instructing the Council to consider the question of prizes, and report thereon at the next annual meeting; this action, however, not to militate against any arrangements for the presentation of prizes next year if in accord with the vote of the Association in 1887.

On motion of Prof. Good it was *Resolved*, That the American Pharmaceutical Association desires to record its appreciation of the ethical position taken by the American Medical Association at its last meeting in its efforts to discourage the use of secret remedies and traffic in nostrums.

Resolutions of thanks to the local secretary and the various committees were offered by M. Alexander, and adopted.

Standing and in silence the association adopted resolutions of sympathy at

the sudden illness of Professor Bedford. In another place we record his death, which occurred on the Wednesday following.

After installing the newly-elected officers, and reading the minutes of the last session, the Association finally adjourned to meet next year in Chicago, on the third Tuesday of August, 1893, unless the time be changed by the Council.

The date for the meeting of the Seventh International Pharmaceutical Congress at Chicago has been fixed for the fourth Tuesday of August, 1893.

During the days of their stay at the Profile House the members visited the places of interest in the neighborhood. Within easy walking distance is the "profile," a stone face formed on a spur of the mountain by three ledges of granite, nearly forty feet in height, and overlooking the placid Profile Lake, twelve hundred feet beneath it; and in the opposite direction Echo Lake. The Basin, Pool and Flume are distant four and six miles, and a visit to Sugar Hill requires a drive of about sixteen miles. Ascents of Mounts Lafayette and Cannon were also made by a number of the party. On three evenings readings were given by Professor Churchill, and music had been provided in the lobby or the parlors of the hotel afternoons and evenings.

On Tuesday morning, July 19, about one-half of the party left by rail at 7.30, visited Mount Washington, took dinner at the Summit House and then returned, passing the evening and night at the Crawford House. The remainder of the party started about three hours later, passed the night at Fabyan's, and reached the Crawford House on Wednesday morning. From here the ascent of Mount Willard was made, from the summit of which a grand view is had of the upper Saco Valley. Dinner was provided at the Crawford House, the excursionists being the guests of Vermont druggists, Wells, Richardson & Co., Burlington, and after dinner the party left by rail, some returning to Boston, but the large majority, accepting the invitation extended by Maine, proceeded to Portland, arriving there early in the evening, and put up at the Falmouth, United States, Preble and other hotels. At the former of these an informal reception was held, at which many of the pharmacists, druggists, physicians and other citizens of Portland were present. The pharmacists of the city, joined by the Board of Trade and others, provided a carriage ride for Thursday morning, thus affording the visitors an opportunity for seeing the principal portions of the city and some of the suburbs. The drive terminated at a steamboat landing, where a steamer was in waiting to convey the visitors and their hosts on an excursion through the harbor and a part of Casco Bay, which is studded with a large number of islands, big and little. Finally, Long Island was reached, where a clam-bake was in progress and dinner was served. The return trip to the city was made in time to take the evening trains for Boston. But many preferred remaining in the hospitable city over night, while another large party secured state-rooms aboard the steamer *Portland* for a sea voyage to Boston during the evening and night. The latter city, which had been the gathering point of the party nearly two weeks earlier, became now the dividing point on the homeward trips.

Aside from the work done at the sessions, the meeting was a memorable one for the historical places visited, for the sublime scenery beheld, for its social features and for the unbounded hospitality tendered by Massachusetts, New Hampshire, Vermont and Maine. But a shadow was cast at its close by the sudden removal of one of the party from among the living.

# OBITUARY.

*Joseph C. Turnpenny*, deceased at his residence, No. 813 Spruce Street, Philadelphia, June 15, 1892, in the 80th year of his age. Joseph was the youngest of the four children of John and Tabitha Turnpenny. He was born in Sheffield, England, September 28, 1812. His father was a merchant in Sheffield. Joseph and his elder brother Frederick were the only children who lived to come to the United States, about the year 1814. Frederick studied medicine, and had the degree of M. D. conferred on him by the University of Pennsylvania; he deceased in 1840. Joseph received his early education at the School of the Society of Friends on Pine Street near Second Street. In 1828 he was apprenticed to Henry M. Zollickoffer, whose store was at Sixth and Pine Streets, to learn the business of an apothecary. He graduated in the class of 1833 at the Philadelphia College of Pharmacy, and went into business at the N. E. corner of 10th and Spruce Streets, in 1834. In 1864 he retired from business and was succeeded by Samuel S. Bunting. He was elected to membership in this College in 1834, and served the college as its Treasurer for a number of years (1844 to 1853), and also as a member of its Board of Trustees. His name is attached as author, or as joint-author with that of the late Aug. Duhamel, or with Wm. Procter to several papers on practical pharmaceutical subjects, which were published in the 14th and 17th volumes of this journal.

In November, 1853, he married Elizabeth Richardson, daughter of John and Sarah Richardson, of New Castle County, Delaware.

As a member of the religious society of Friends he maintained through life their customs of dress and address. For many years he was an active member of many of the charitable institutions of this city, and devoted much time to the alleviation of the necessities of the poor and suffering. His long service in the Board of Managers of the Pennsylvania Hospital terminated only with his life. Affable in his disposition and courteous in his address, carefully correct in all of his business transactions, he leaves an honored name on the roll of the deceased members of this College. His health was much impaired several years ago by an attack of pneumonia, and to that trouble he finally succumbed. His wife, who had been an invalid for some time, deceased a few days after her husband. They leave no children. C. B.

*Peter Wendover Bedford*, Ph.G., Emeritus Professor of Pharmacy of the New York College of Pharmacy, died suddenly at the Profile House, N. H., July 20, in the fifty-sixth year of his age. He was born August 1, 1836, in Johnsville, Dutchess Co., N. Y., and was the eldest of eight children. He received his early education at a private school at Mount Vernon, and when only twelve years old became an apprentice in a pharmacy on Bleecker Street, New York, and subsequently while engaged with Mr. Ewen McIntyre, attended lectures at the New York College of Pharmacy, from which institution he graduated in 1858. Shortly afterwards he went into business at 769 Sixth Avenue, New York, and also conducted a branch store at Mount Vernon, N. Y. Soon after becoming a member of the New York College of Pharmacy, he was chosen secretary in 1860, and later on one of the trustees. From about 1865 he had occasionally given instruction and sometimes lectured to members of the classes of that College, and in 1873 he was elected professor of pharmacy

which position he held until 1891. His connection with the American Pharmaceutical Association dates from the year 1859, and in the following year he contributed to its Proceedings a paper on the depreciation of Smyrna opium in medicinal value, which marks the beginning of his literary career. Having given up his retail stores in 1870, he became connected with wholesale houses, first with Tarrant & Co., and subsequently with Lazell, Marsh & Gardner, until 1883. The excellent opportunities afforded him through this connection widened his views on commercial subjects, and his writings became more varied and more frequent, more especially his contributions to the *Druggists' Circular*, then edited by Dr. Newton, and his subsequent papers for the *Pharmaceutical Record*, the editorship and management of which he undertook in 1883, at the same time changing its former title of "Martin's Chemists' and Druggists' Bulletin." It was due mainly to his energy that the New York State Pharmaceutical Association was organized in 1879; Prof. Bedford was complimented by being elected its first president. In 1884, he became a member of the New York City Board of Pharmacy, and was elected its president, continuing in service until the time of his death. In the American Pharmaceutical Association, besides having served on many committees, he was corresponding secretary from 1860 to 1862, and again from 1863 to 1866, when that office was discontinued, he having served as recording secretary at the meeting in Philadelphia in 1862, and at the first session of the Baltimore meeting in 1863. He was the first local secretary elected by the association and acted as such for the New York meeting in 1867. In 1881, at the Kansas City meeting, he was called to the presidential chair. He was very rarely absent from the annual meetings of this association during a period of thirty-two years.

Professor Bedford was, verily, a busy man. His genial disposition secured for him a large circle of friends; and it will, indeed, be impossible to fill his place in the varied spheres alluded to above. Though retired as a teacher, he retained until the last his interest in the promotion of pharmacy, and his devotion to the institutions and bodies with which he had been connected during his busy life. For some time past he had been aware of some affection of the heart; but during the trip on the steamer *Puritan* from New York to Boston, he appeared to be in his usual good health and spirits. He was somewhat indisposed on the journey to the White Mountains, but was taken seriously ill on the night of July 16, his condition being aggravated by apoplexy on Monday, until he died on the following Wednesday. His body was taken to his home in New York, where the funeral services were held on Friday, July 22.